



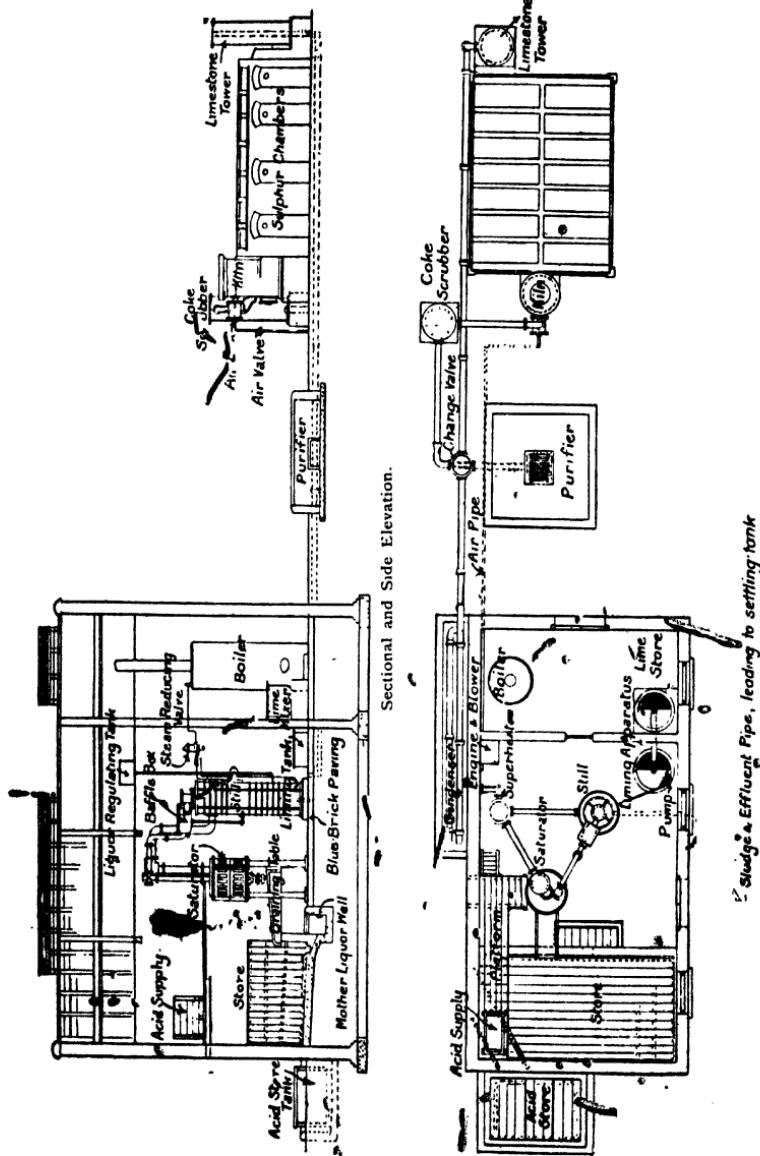




THE MANUFACTURE OF  
SULPHATE OF AMMONIA  
AND CRUDE AMMONIA







**Sulphate of Ammonia and Clays Sulphur Recovery Plant at the Wrexham Gasworks.  
PLA.N.**

# THE MANUFACTURE OF SULPHATE OF AMMONIA AND CRUDE AMMONIA

BY  
GASCOIGNE T. CALVERT

SECOND EDITION  
REVISED AND ENLARGED



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## PREFACE TO FIRST EDITION

"Of the making of books there is no end," and yet, amid the multitude of them, the proprietors of THE GAS WORLD feel that there is still a need for a practical handbook upon "The Manufacture of Sulphate of Ammonia," a handbook which, while it will be of interest to the large manufacturers, will yet be of such modest dimensions as to be within the reach of the small manufacturer with limited means, and which, while it will contain full working details, shall yet be written in such simple language that it may be easily understood by any workman of average intelligence—in short, not so much the work of the scientist or the man of high literary attainments as that of a practical works' man writing for his fellow-workers. Such a work the proprietors of THE GAS WORLD asked the writer to undertake, and after much hesitation and mature consideration he agreed to do so; and he now therefore makes his bow to that section of the British public who are interested in the subject under discussion.

In a work of this character space prevents our dealing with more than just a typical example or two of each kind of plant, and the omission from these pages of mention of any maker's plant must not, for one moment, be taken to infer that such a plant is unworthy of notice or inferior to the examples selected.

The writer desires to thank most heartily the various manufacturers mentioned in these pages for the kindly manner in which they have responded to his requests for particulars and drawings of their plants, etc.; in fact, the response has been so generous that out of the mass of valuable material furnished it has been, in many cases, most difficult to make a selection,

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as he felt that, had space permitted, he should have liked to use all.

He also desires to thank Mr OWEN EVANS, assistant manager of the Wrexham gasworks, for kindly giving permission to reproduce the drawing of their sulphate of ammonia plant as frontispiece; and also Mr PHILIP G. G. MOON, of the Bournemouth gasworks, for his kind permission to reprint his valuable paper on "Sulphate Manufacture for Small Works," reproduced in Chapter XIV. of this work.

In conclusion, the author desires also to thank his colleagues in the works—Messrs JOHN WYLD, WILFRID WYLD and W. A. TWINE—for the valuable assistance they have rendered him in the preparation of the work.

G. T. C.

ERDINGTON,  
NEAR BIRMINGHAM,  
*September 1911*

## PREFACE TO SECOND EDITION

THE kindly reception afforded to the first edition of this work, the fact that that edition is now out of print, and the numerous inquiries that reach the publishers for a second edition, must be the writer's excuse for venturing out of his retirement to prepare this second edition.

To the many friends who have so kindly assisted him to bring the matter up to date he tenders his heartfelt thanks, and if his readers profit as much by the perusal of his work as he has had pleasure in writing it, he will be quite satisfied with the results.

G. T. C.

•  
•  
ERDINGTON,  
*September 1917*



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# THE MANUFACTURE OF SULPHATE OF AMMONIA

## CHAPTER I

### SULPHATE OF AMMONIA: ITS COMPOSITION AND ANALYSIS

SULPHATE of ammonia is a salt formed by the distillation of ammonia gas into sulphuric acid. It has the following formula :—



In a perfectly pure salt entirely free from moisture this would give a composition of 25.757%  $\text{NH}_3$  and 74.243%  $\text{H}_2\text{SO}_4$ . This state of purity, however, is never met with on a large scale.

The commercial article is a salt of light grey colour, and is guaranteed to contain a minimum of 24%  $\text{NH}_3$ . It usually contains from 24.25% to 24.75%  $\text{NH}_3$ , from 2 to 4% of moisture and from 0.15% to 0.4% uncombined  $\text{H}_2\text{SO}_4$ . The buyers also frequently stipulate that it shall be free from cyanides, as these are deleterious to vegetable growths. As the greater part of the sulphate of ammonia produced is used as a chemical manure, either by itself or combined with other substances, the necessity for this freedom from cyanides will be readily understood.

In works where the storage capacity is ample, it sometimes happens that the sulphate of ammonia is kept for a rather long time, and in this case, especially if the salt has been turned over occasionally, it loses so much moisture that it frequently contains from 24.75% to 25.25%  $\text{NH}_3$ . In works where the sulphate of ammonia is dried in a hydro-extractor, the same result may be obtained by running the extractor a little longer. Such a salt, especially if also of fairly white colour, usually fetches the top market price, but the little extra price secured scarcely pays for the extra ammonia above the 24% guaranteed in the normal way.

From time to time efforts have been made to adopt the practice of selling upon the basis of so much per cent. of  $\text{NH}_3$ ; for example, if 24% was sold at £12 per ton, then 24.4% would be  $\frac{12 \times 24.4}{24} = £12.48$  per ton. Such a method would certainly be fairer to the manufacturer, but unfortunately, for

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some reason, up to the present these efforts have not been attended with success.

*Sampling.*—The usual method of sampling a parcel at the works is to take a small pinch from each bag as it is packed, place it in a large jam jar or other suitable vessel with a lid, and when the packing of the parcel is completed intimately to mix the contents of the jar, and then from this intimately-mixed sample to fill, say, three 8-oz. wide-mouthed bottles, taking care to cork, seal and label them immediately afterwards. One of these samples is afterwards used for analysis and the other two reserved for reference in case of any dispute arising.

In the event of the packing of the parcel extending over more than one day, it is desirable each evening thoroughly to mix up the contents of the jar and afterwards fill a glass bottle of, say, 1 lb capacity, carefully either corking and sealing same, or—which is more convenient—using an india-rubber stopper to cork the bottle. When the parcel is completed these large samples are carefully mixed together in proportion to the weights they represent, and from this mixed sample the three smaller samples are taken, as in the previous case.

In sampling from a large heap it is desirable to dig into the heap for about a foot, and to take the sample from the bottom of the hole thus formed, repeating this in about a dozen places, taking care that these samples are taken at different heights in the heap. The samples thus taken are carefully mixed together and the three bottles filled, as in the previous case.

*Analysis.*—The sample of sulphate of ammonia is usually analysed for ammonia ( $\text{NH}_3$ ), moisture and uncombined sulphuric acid, the two first-named being of most importance.

For the ammonia test, two standard solutions are necessary, viz.:—normal sulphuric acid (*i.e.*, a solution containing 49 grammes of pure  $\text{H}_2\text{SO}_4$  per litre) and normal sodium hydrate (a solution containing 40 grammes of pure sodium hydrate per litre).

To make the former, into a Winchester quart bottle put two litres of water and add to it slowly about 60 c.c.'s of sulphuric acid (the quality known as R.O.V. or D.O.V. will be sufficiently pure for the purpose). Shake up well and allow to cool. While this solution is cooling, a small quantity of carbonate of soda is prepared for standardising, by being heated in a platinum or porcelain crucible over a low bunsen flame, the flame only being of such a height as just to touch the bottom of the crucible. After heating for about a quarter of an hour the sodium carbonate is allowed to cool under a desiccator, ready for weighing. Messrs J. W. Towers & Co., Limited, supply a quality of carbonate of soda "Puriss. Anhydrous" made for this special purpose. To standardise the acid, a 50 c.c.

burette is filled with the acid solution, after being carefully rinsed out with the same. 2.65 grammes of the dried sodium carbonate is carefully weighed and transferred to a porcelain evaporating dish of about 9-inches diameter. A little water is added and a little strong solution of methyl orange (this being used as the indicator of acidity or alkalinity). The acid is slowly run from the burette into the solution until, after standing for a few moments, the solution is neutral between pink and yellow, when the number of c.c.'s used is read off. If the acid solution is quite normal the number of c.c.'s required will be exactly 50, but as the solution has been made rather stronger than this to allow for impurities in the acid used, it will probably have taken a few c.c.'s less than this quantity. Supposing it has taken 46 c.c.'s, it will be obvious that for every 46 c.c.'s it will be necessary to add to the solution an additional 4 c.c.'s of water. After doing so and allowing to cool again, a similar test with the sodium carbonate is again made, and this time the 2.65 grammes of sodium carbonate should require just 50 c.c.'s of acid solution to neutralise it.

The solution of methyl orange referred to is made by adding distilled water to the methyl orange powder until the solution is of a good strong colour. This solution is much more reliable as an indicator than litmus solution.

To make the normal solution of sodium hydrate, about two litres of water are placed in a Winchester quart bottle and about 85 grammes of sodium hydrate (in sticks) slowly added to same. After the sticks are dissolved, shake up well and allow to cool. The solution is now ready for standardising. To do this, fill the burette already used with normal sulphuric acid, and a second 50 c.c. burette with the solution of sodium hydrate solution. Into a dish run 20 c.c.'s of the normal acid, add a small quantity of methyl orange solution, and then run into it the sodium hydrate solution until the acid is just neutralised. Repeat the experiment, and if the second experiment confirms the first they may be taken as correct. It will be found that the 20 c.c.'s of acid have scarcely taken 20 c.c.'s of the soda solution, and again it will be necessary to add the necessary quantity of water to the soda solution to make up the deficiency. After doing so check the finished solution, and if now 20 c.c.'s of acid require 20 c.c.'s of the sodium hydrate solution to neutralise them, the solution may be taken as correct.

In order to avoid confusion it is desirable to have the burette for the normal sulphuric acid with a glass tap and the one for the normal solution of sodium hydrate with the usual india-rubber tube, glass-pointed tube and brass clip, the two being held in a suitable double burette stand.

A very suitable apparatus in which to make the test is

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shown in Fig. 1. *a* is a flask of heat-resisting glass of about  $1\frac{1}{2}$  litres' capacity, having an india-rubber stopper with two holes. Through one of these is inserted the separator marked *b*, of about 50 c.c.'s capacity, and having a glass tap as shown. Through the other hole is inserted a leading tube with two small bulbs in it, *c*. By india-rubber tubing this is connected

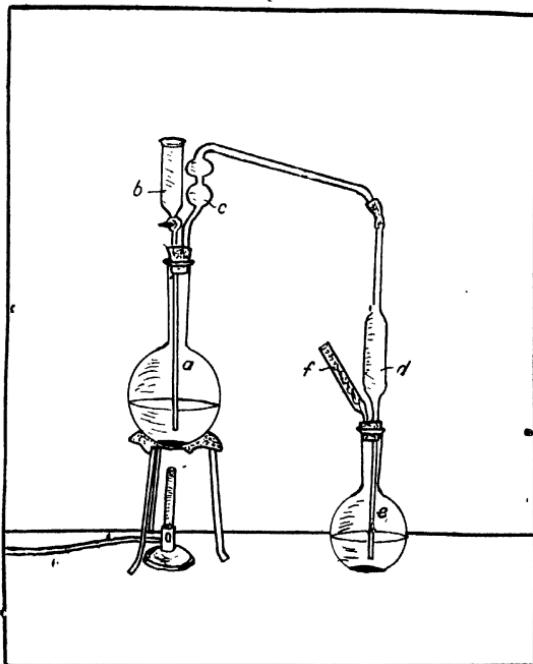


FIG. 1.—Sulphate of Ammonia Testing Apparatus.

to the inlet tube, *d*, of the second flask. This is formed from a pipette of about 100 c.c.'s capacity, and it is inserted through one of two holes in the india-rubber stopper of the second flask, *e*. This flask is about 700 c.c.'s capacity. In the second hole a calcium chloride tube, *f*, is placed, the small tube at the bottom having been previously bent at a slight angle as shown. This tube should be filled with broken pieces of glass. The object of the tubes, *c* and *d*, is to prevent danger of the contents of the flask, *e*, being syphoned back into the

flask, *a*, through draughts in the laboratory, while the object of the calcium chloride tube, *f*, is to catch any slight splashes that may be thrown off from the flask, *c*.

To make the tests, the sample of the sulphate of ammonia is ground up in a mortar, thoroughly mixed up, and three samples of  $2\frac{1}{2}$  grammes each carefully weighed out on the balance.

The first sample is washed into the flask, *a*, through a glass funnel, with water. Into the flask, *c*, is carefully measured 40 c.c.'s of the normal sulphuric acid and a little distilled water added. The apparatus is now connected up, and through the separator, *b*, is added about 50 c.c.'s of strong sodium hydrate solution, care being taken to close the tap as soon as the solution has passed through. A little water is then run through the separator to clear the tap, the bunsen burner is lighted, and the distillation carried on for about an hour. At the close of this time, on removing the stopper from the flask, *a*, there should be no smell of ammonia, nor should the steam issuing from it turn a litmus paper blue. Instead of removing the stopper from the flask, *a*, the apparatus may be conveniently disconnected between *c* and *d*. The contents of the flask, *c*, are now allowed to cool (this may be accelerated by allowing cold water to run over it), and the contents are then transferred to a porcelain dish. Methyl orange solution is then added, and normal sodium hydrate solution run in until the contents of the dish are just neutral, this being shown by the colour of the solution being neither distinctly pink nor yellow. A reading of the number of c.c.'s of the normal sodium hydrate required to achieve this result is carefully taken, and this quantity is deducted from the 40 c.c.'s of normal sulphuric acid originally taken. The difference is multiplied by 0.017 (this being the quantity of ammonia that 1 c.c. of either the acid or soda solution is equivalent to), and this result again multiplied by 40, when the result will show the percentage of  $\text{NH}_3$  contained in the sulphate of ammonia.

The following example will make this clear :—

Normal sulphuric acid				
taken	•	•	•	40 c.c.'s
Normal sodium hydrate				
solution required to				
neutralise	•	•	•	4.0

$$\text{Difference} \quad . \quad 36.0 \times 0.017 \times 40 = 24.48\% \text{ NH}_3.$$

To ascertain the moisture the second  $2\frac{1}{2}$  grammes is taken and dried in a hot-water bath for about four hours. It is then re-weighed and the weighing again booked. It is put back for

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another half hour and again weighed. If the third weighing is the same as the second it is thus found to be perfectly dry; if not it is again put back and allowed to remain until the weight is constant. The difference between the original weight and the weight when dry will, of course, represent the amount of moisture present, and this multiplied by 40 will give the percentage. Example :—

	Grammes.
2½ grammes of sulphate ammonia and watch glass . . . . .	12.500
Second weighing, say, 12.440	
Third weighing 12.425	
Fourth weighing . . . . .	<u>12.425</u>
Loss . . . . .	0.075 × 40
	= 3.00% moisture.

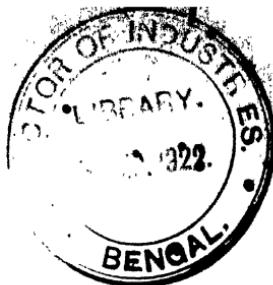
To ascertain the amount of uncombined (or free) acid present, the third 2½ grammes is dissolved with distilled water in a porcelain evaporating dish and neutralised with the normal sodium hydrate solution from the burette, methyl orange again being used as the indicator. The quantity of sodium hydrate solution used is multiplied by 0.049 (this being the amount of sulphuric acid that 1 c.c. of the normal sodium hydrate solution is equivalent to) and again multiplied by 40, when the result will show the percentage of the uncombined acid present in the sulphate of ammonia.

Example :—

Quantity of normal sodium hydrate required —

$$0.2 \text{ c.c.} \times 0.049 \times 40 = 0.392\% \text{ free H}_2\text{SO}_4.$$

For this last test, if greater delicacy of testing is required, it is advisable to make up another standard solution of sodium hydrate by taking 100 c.c.'s of the normal solution and adding to it 900 c.c.'s of water. The strength of the solution is then spoken of as deci-normal ( $\frac{N}{10}$ ) and 1 c.c. is then equal to .0049% of sulphuric acid only. The calculation would then require to be, of course, altered accordingly.



## CHAPTER II

### THE RAW MATERIALS: AMMONIACAL LIQUOR, SULPHURIC ACID AND LIME

*Ammoniacal Liquor.*—Practically the whole of the sulphate of ammonia produced is made from ammoniacal liquor obtained from the following sources:—

- (a) Gasworks.
- (b) Coke-Oven Plants.
- (c) Shale Works.
- (d) Blast Furnaces in Iron Works.

From a practical standpoint the liquors from the first three of these sources present many points of similarity; they all contain ammonia,  $\text{CO}_2$  and  $\text{H}_2\text{S}$ , and differ chiefly in their strength, that produced in gasworks usually being the richest in ammonia, while that produced from coke-oven plants contains only about half the quantity of that from the gasworks, and that produced from shale works is even weaker still. In the annual report of the Chief Inspector under the Alkali, etc., Works Act, for 1896, much interesting matter regarding ammoniacal liquor is given and in a table in the Appendix are given comparisons of the constituents of the different types of liquor, the information for these being extracted from the Alkali Inspector's report above mentioned.

In passing, we may say that the sulphate of ammonia industry is deeply indebted to Mr Linder, one of the assistants of the Chief Inspector, for the many careful analyses of ammoniacal liquor which he has made during the last few years, and the deductions which he has drawn from them, which have been published in the Chief Inspector's reports for the years 1903-4-5 and 6, and which will well repay perusal.

Ammoniacal liquor is a very complex substance, the ammonia existing under various combinations. For practical purposes, however, they are classed under two divisions:—

- (a) Volatile, or as it is frequently, but erroneously, called “free”—that is, those combinations of ammonia that can be volatilised by the application of steam, and,
- (b) Those combinations of ammonia that require the addition of an alkali such as lime to liberate them.

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Under the former heading is classified :—

Free ammonia (the presence of ammonia in the free state in ammoniacal liquor is exceedingly doubtful, and, if it exists at all, it can only be in very newly made liquor)

Under the latter heading is classified :—

Ammonium sulphate	Ammonium thiocarbonate
," sulphite	," sulphocyanide
," chloride	," ferrocyanide.
," thiosulphate.	

## ANALYSIS

*Specific Gravity.*—The specific gravity of ammoniacal liquor is usually expressed in degrees Twaddel (Tw.) at a temperature of 60° Fahr. This is ascertained by means of the hydrometer, the most suitable form for this purpose being one made with a large bulb and the scale marked in  $\frac{1}{4}^{\circ}$  from 0° to 10° Tw. As very few hydrometers are absolutely correct, it is always advisable, before putting a new one into use, to check its correctness by means of the specific gravity bottle.

To do so, first weigh a 50-gramme specific gravity bottle on the balance to ascertain its tare. Then fill with distilled water at  $60^{\circ}$  Fahr. and weigh again. The difference will give the weight of water actually contained. This precaution is very necessary, as it is rare to find that the bottle actually contains exactly the 50 grammes marked on it. The bottle is then rinsed out with the ammoniacal liquor to be tested, filled—the temperature being exactly  $60^{\circ}$  Fahr.—and then reweighed. The difference between this last weight and the tare of the bottle will, of course, give the weight of the liquid being tested, and if this be divided by the weight of the water actually contained in the bottle, the result will give the specific gravity of the liquor. The following example will make this clear:—

	Grammes.
Weight of specific gravity bottle and distilled water	75.917
Weight of specific gravity bottle empty	25.926
Weight of water	49.991
Weight of specific gravity bottle full of ammoniacal liquor being tested	77.516
Weight of empty specific gravity bottle	25.926
Weight of ammoniacal liquor	54.590

$51.590 \times 1000 \div 49.991 = 1031.9$ . The specific gravity of the ammoniacal liquor being tested is therefore 1031.9, taking water as being 1000. To ascertain from this the strength that the Twaddel hydrometer should show, deduct from the specific gravity shown 1000 and divide the remainder by 5, when the result will be the  $^{\circ}\text{Tw}$ . In this case the calculation will be

$$\begin{array}{r}
 1031.9 \\
 \text{less } 1000 \\
 \hline
 5) \quad 31.9 \\
 \hline
 6.38
 \end{array}$$

6.38  $^{\circ}\text{Tw}$ . is the correct reading, and a comparison of the actual reading on the hydrometer will show the correction to be made in future readings of the hydrometer at that strength. If this is done with the various strengths of liquor likely to be tested with it, and a scale of corrections prepared, it will remove one of the most fruitful sources of difficulty in agreeing upon the strength of liquor.

*Ounce Strength.*—The term "ounce strength" is applied to the number of ounces of pure sulphuric acid required to neutralise 1 gallon of the ammoniacal liquor being tested, and the test as formerly made formed a rough-and-ready method of gauging the strength and value of the liquor. The test, however, as made only took into account the volatile ammonia, and the point when neutrality was reached was anything but easy to determine.

It is therefore much better to adopt the modern method of distilling for total ammonia, which will shortly be described, and multiply the result by 4.61, when the result will be the accurate ounce strength.

Generally speaking, the ounce strength is, roughly, about double the  $^{\circ}\text{Tw}$ . strength; but it will be readily seen that with a substance so variable in its composition as ammoniacal liquor, such a statement can only be taken as being very approximately correct.

*Total Ammonia.*—For the purpose of testing the total amount of ammonia present, the apparatus shown in Fig. 1, in the preceding chapter, may be employed with advantage.

10 grammes of the ammoniacal liquor to be tested is carefully weighed on the balance, in a small beaker, and transferred to the flask, *a*. A little water is added, 15 c.c.'s of the normal sulphuric acid are placed in the flask, *e*, and a little water also added. About 10 c.c.'s of strong sodium hydrate solution are then run through the separator, *b*, and this is then rinsed out with water. The remainder of the operations are then exactly the same as in testing for ammonia in the sulphate of ammonia,

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the only difference being in the calculation. The following example will make things clear and will also give the necessary calculation :—

10 grammes ammoniacal liquor taken.

Normal acid used	15.0
Normal sodium hydrate used	2.1

Difference .  $12.9 \times 0.017 \times 10 = 2.193\% \text{ NH}_3$   
present in the sample.

Instead of weighing the 10 grammes, 10 c.c.'s may be measured from a pipette—care being taken that the capacity of the pipette is as shown on it, or a suitable correction made—and the result will then require a correction as follows :—

The result will require to be multiplied by 1000 and dividing by the specific gravity of the liquor (this being obtained by taking the strength shown by the hydrometer, multiplying by 5 and adding 1000).

Example :—

10 c.c.'s ammoniacal liquor taken showing 5° Tw. at 60° Fahr.

Normal acid taken	15.0
Normal sodium hydrate taken	2.1

Difference .  $12.9 \times 0.017 \times 10 = 2.193 \text{ grammes}$   
 $\text{NH}_3$  in the 10 c.c.

Specific gravity 5° Tw.  $\times 5 + 1000 = 1025$ :

$2.193 \times 1000 \div 1025 = 2.139\% \text{ NH}_3$  present in sample.

*Volatile Ammonia.*—The test for this is made in exactly the same manner as for the total ammonia, except that in this case no sodium hydrate solution is added to the contents of flask, *a*. Care should be taken that no soda is left in the flask from a previous distillation. Common hydrochloric acid (spirits of salts) will be found very useful for cleaning out the flasks.

*Fixed Ammonia.*—By subtracting the volatile ammonia from the total ammonia the amount of fixed ammonia is readily obtained.

*Sulphuretted Hydrogen.*—To test for this substance a solution of ammonia-cuprous sulphate is required. This is made by dissolving, say, 25 grammes of pure copper sulphate in nearly a litre of water and adding ammonium hydrate until the precipitate first formed is re-dissolved and then making up the solution to exactly one litre.

To standardise this solution, take 20 c.c.'s of it and precipitate the copper with potassium hydrate (the quality known as "pure by alcohol") solution. Transfer this precipitate on to a filter paper and thoroughly wash with distilled water until neutral. Then dry the precipitate on the filter. Tare a porcelain crucible and ignite the precipitate over it, transfer the ignited precipitate to the crucible and ignite at a low heat for some time, adding a single drop of pure nitric acid to the ash. Cool under a desiccator and weigh again. The difference will be the weight of copper oxide ( $CuO$ ) present in the 20 c.c.

Grammes  $CuO \times 32 \div 20 \div 79.12 =$  value per c.c. of the solution expressed as equivalent to sulphur in the  $H_2S$ .

In obtaining the tare by the above method, a dry filter paper should be burned and ignited in the crucible in the same manner as the precipitate is treated, and included in the weight of the crucible.

To test for  $H_2S$  in the ammoniacal liquor, 10 grammes are taken, placed in a porcelain evaporating dish and diluted with a little water and a little .880's ammonia. The copper solution is placed in a 50 c.c. burette and slowly run into the liquor in the evaporating basin until the precipitate formed begins to coagulate and a very slight blue colour is observed for a slight distance round the edge. The number of c.c.'s used is then read off,  $\times$  by the value per c.c.  $\times$  10, and the result is the percentage of sulphur present in the liquor as  $H_2S$ .

Example :—

10 grammes of ammoniacal liquor taken.

Copper sulphate solution required 15.5 c.c.  $\times$  .0032 (value per c.c. of solution)  $\times$  10 = 0.496% sulphur as  $H_2S$ .

The other substances present in ammoniacal liquor are not so easy for the beginner to analyse, and a full description of them would take up too much room in our handbook. Should the reader desire to make a complete analysis of the constituents of the liquor, he is referred to the fortieth report of the Chief Inspector under the Alkali, etc., Works Act, for the year 1903, pages 31-39, where Mr Linder gives full particulars for the necessary work in a very clear and concise manner.

*Sulphuric Acid.*—This is a liquid of heavy density having the following formula :—



which combines with many bases, forming a series of well-defined salts. To the touch it has a somewhat oily feel, hence its popular name of "oil of vitriol." It is intensely corrosive and must therefore be handled with care.

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It is made from four different sources of sulphur, viz. :—

- (a) Native brimstone or recovered sulphur.
- (b) Spent oxide.
- (c) Pyrites.

Of these the sulphuric acid made from native brimstone or from the "recovered sulphur" from the alkali process is best, but it is too expensive for sulphate of ammonia making and is used only for laboratory purposes or for other purposes where great purity is required.

The acid made from spent oxide is very suitable for sulphate of ammonia manufacture, and as most sulphate of ammonia manufacturers are more or less intimately connected with the gas industry, they will no doubt have a leaning to that source which is connected with that class of works.

The acid made from pyrites is generally known as "stone acid," and is divided into two qualities, viz. :—de-arseniated and ordinary. So long as it is fairly free from arsenic it is suitable for the manufacture of sulphate of ammonia, but should arsenic be present in appreciable quantity, the sulphate of ammonia produced from it is of a more or less brown colour, and does not usually sell at so high a price.

Each kind of acid is supplied in three different qualities, viz. :—

- (a) R.O.V. (rectified oil of vitriol) or D.O.V. (double oil of vitriol), both names being used for the same quality of acid. This quality of acid is prepared by heating in either retorts or dishes and is the commercially pure article. It has a specific gravity of 1.845 taking water as 1.000, this being equivalent to 169° Tw. at 60° Fahr. This quality is too expensive to use in sulphate of ammonia manufacture.
- (b) B.O.V. (brown oil of vitriol). Where acid has to be purchased this is the quality of acid that is usually used in the manufacture of sulphate of ammonia, in order to save carriage. In winter it is usually supplied of a strength ranging from 1.720 to 1.730 specific gravity (144° to 146° Tw. at 60° Fahr.), as if it exceeds the higher strength its liability to freeze is much greater. In summer the usual strength supplied ranges from 1.730 to 1.740 specific gravity (146° to 148° Tw. at 60° Fahr.), as at that season there is no danger from frost.
- (c) Chamber acid. This acid generally ranges in strength from about 1.500 to 1.600 specific gravity (100° to 120° Tw. at 60° Fahr.), and if the sulphate of ammonia is made in the works where the sulphuric acid is also

made, this is a very convenient form in which to use it, as it saves the cost of concentrating the acid to the B.O.V. strengths.

A table will be found in the Appendix giving the percentage of real acid contained in dilute acid of the various strengths.

The acid supplied to the sulphate of ammonia manufacturer is usually of a brown colour, and if placed in a glass jar in small quantities, the best acid shows only a very slight trace of this colour. It should be fairly clear and should not show more than a minute trace of nitrous compounds.

*Lime.*—As mentioned under the heading of "Ammoniacal Liquor" the non-volatile ammonias present require the addition of lime in the form of milk of lime, as well as steam, in order to expel them from the liquor. For this purpose the writer has used lime from Silverdale, in the Furness district, from the Skipton district in Yorkshire, from the Buxton district, from the Clay Cross district in Derbyshire, and from the Dudley district near Birmingham, and no doubt many similar limes may be obtained in various parts of the country. The lime supplied from each of the above districts is very similar in quality, and the following analysis (made in the laboratory of the Nечells Chemical Works, Birmingham) may be taken as typical of any of them :—

Total lime (estimated as CaO) (of this quantity 92.4% represents the quantity actually exist- ing as CaO, this being the effective portion) . . . . .	95.4
Iron and alumina . . . . .	.94
Silica . . . . .	1.14
Water and carbon dioxide . . . . .	2.52
	<u>100.00</u>

The analysis of lime is rather beyond the tyro in chemistry, but should he desire to try, he is referred to more pretentious books than the present one for full particulars.

The sampling is somewhat difficult, and should be carried out in a manner similar to that in which a truck of coal is sampled for experimental purposes. As a truck of lime is being emptied a little should be broken off a lump occasionally and a fair proportion of any small that may be present added. When the whole of the samples have been drawn they should be intimately mixed and about a bucketful removed, the rest being discarded. The bucketful should then be broken up into small lumps, intimately mixed and about 1 lb. taken, the rest again being discarded. This pound should then be coarsely ground in a mortar, again intimately mixed and about 4 ounces taken.

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and ground to very fine powder. This will then be the sample for analysis. Even after all these precautions have been taken, it may happen that, through an undue proportion of unburnt lime having been included, the sample does not fairly represent the bulk, and may be very misleading to the purchaser. The writer remembers a case where, after taking all the above precautions, a sample showed only about 65% of CaO, whereas there was every reason to believe that the bulk was of the average quality, containing about 92% of CaO.

Fortunately there is a very ready method of ascertaining the approximate purity of the lime being used, viz.—to weigh the lumps of undissolved lime removed from the lime mixers and to calculate the % they represent on the lime used. During ~~work~~ period of six months, in the works with which the writer was connected, this undissolved lime represented 2.25% of the lime used, and during another similar period 2.30%. In ordering the lime it should be specified to be best hard burnt and hand picked, and should the proportion of insoluble lime rise much above the quantity indicated above, there is just cause to complain that the lime is insufficiently burnt.

If lime is kept for some time and allowed to "fall" its efficiency for sulphate of ammonia manufacturing purposes is decreased, so that it is obvious that it should travel in well-sheeted trucks and be stored under cover in a dry place.

If too much alumina is present in a lime there will be a tendency to its becoming pasty and not travelling so far down the still, thus tending to choke up the still more rapidly and also not liberating the non-volatile ammonias so readily.

## CHAPTER III

### PLANT REQUIRED FOR THE MANUFACTURE OF SULPHATE OF AMMONIA : GENERAL DESCRIPTION—SUPPLY OF AMMONIACAL LIQUOR—TACHOMETER—SUPERHEATERS

WE will now proceed to describe the plant which is necessary for the manufacture of sulphate of ammonia.

The frontispiece shows the plant in use at the Wrexham gasworks. This plant includes provision for dealing with the waste gases either by purification by passing through an oxide of iron purifier or, as an alternative, burning them in a Claus kiln and converting the sulphuretted hydrogen into sulphur, also a settling tank to remove the bulk of the spent lime before the waste liquor is allowed to enter the sewer.

It will be seen that this plant consists of a suitable tank for supplying the ammoniacal liquor to the still, a superheater for heating the liquor previous to its entering the still, the still with its liming arrangements, the saturator, the condensers for further cooling the waste gases after leaving the superheater, so as to enable the oxide of iron purifier or the Claus kiln to deal effectively with them, suitable tanks for storing and supplying the sulphuric acid, a draining table for the sulphate of ammonia as it leaves the saturator, suitable storage accommodation in which to allow the sulphate of ammonia to remain and drain until it is fit to be packed into bags for delivery, a mother liquor tank to collect the drainings from the sulphate of ammonia and thus enable them to be worked up again, a seal pot to prevent the escape of live steam with the waste liquor, a settling tank to remove the excess of solid material before the clear waste liquor is allowed to enter the sewer, and, of course, a suitable steam boiler for raising the steam necessary to effect the distillation of the ammoniacal liquor and to supply the motive power to work the necessary pumps and injectors for the plant.

*Ammoniacal Liquor Supply.*—A very efficient method of supplying the plant with ammoniacal liquor, where the buildings are adapted for its erection, is to provide a cast-iron tank sufficiently high to supply the ammoniacal liquor to the still by gravitation. Such a tank can readily be erected upon girders placed upon the walls of the building, and should be of

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sufficient capacity to hold several hours' supply. If this tank is square or oblong and a float is connected with a gauge placed inside the building, it affords a ready means of gauging the supply to the plant, and the ammoniacal liquor can be pumped up to it at suitable intervals. To prevent the escape of ammonia it is desirable that such a tank should be covered in. A suitable covering consists of 3-inch planks joined with hoop iron tongues, and bolted to the sides of the tank. A small opening in this cover should be left, say a circular hole with a short length of cast-iron flanged pipe bolted over it, to allow for vent when pumping into or feeding the still from the tank.

In cases where such a tank is provided it is desirable to place a small regulating tank on the pipe before it enters the superheater, this tank being controlled by a ball-cock, care

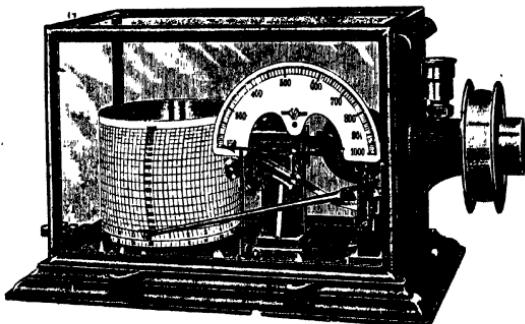


FIG. 2.—Tachometer.

being taken that this ball-cock and its float are made of iron, as the ammoniacal liquor speedily acts upon copper or brass. Such a regulating tank is placed at a sufficient height above the superheater to feed by gravitation, and on the pipe connecting the base of this regulating tank with the base of the superheater is placed a cast-iron gland cock to regulate the supply. If a quadrant is attached to this cock it is of considerable use in regulating. (We would point out that cocks intended for use on ammoniacal liquor should have glands sufficiently deep to take at least two rings of packing, and if the taper of the plug is somewhat great it greatly facilitates the prevention of sticking, and consequent trouble in opening and shutting the cocks.)

In cases where a tank such as has been described cannot readily be erected, the ammoniacal liquor may be pumped directly from the underground or other storage tank into the base of the superheater. When this is done, great care should

be taken to see that the pump works very steadily, as any irregularity of working leads to "high" waste liquor samples and consequent heavy waste of ammonia, if the pump is working too quickly, or in the reverse case, when the pump has slowed down, to an excessive quantity of steam and lime being used unnecessarily.

An instrument of considerable use in regulating the speed of this pump is the tachometer shown in Fig. 2, made by Messrs Schaffer & Budenberg, Limited. This little instrument is worked by means of a light countershaft, a pulley on which is run by a light belt from the flywheel of the pump, and a second pulley and light belt connecting the countershaft to the pulley on the tachometer. On the shaft of the tachometer is a small but heavy brass ring, and the greater the speed at which this shaft is driven the greater will be the tendency of this ring to assume a vertical position. As the ring moves the motion is conveyed to the pointer on the dial and also to a second lever bearing a pen, which records upon a revolving drum the speed at which the pump has worked during the whole of the twenty-four hours. It thus not only indicates very accurately, to the man in charge of the plant, the speed at which the pump is working, but also registers any irregularity, and enables the manager to take the matter up with the defaulting party.

*Superheaters.*—In a sulphate of ammonia plant the superheater is used to raise the temperature of the ammoniacal liquor previous to its entering the still, thus economising fuel and also increasing the capacity of the still to deal with the liquor. For this purpose the waste gases from the saturator are utilised, and as it is necessary that these gases should be cooled in order to fit them for the further treatment mentioned later on, it will be seen that a double object is achieved by the use of a superheater.

The form commonly used is shown in Fig. 3 (page 18). The reader will see that it is practically the familiar "battery" condenser, erected vertically. It consists of a shell and perforated horizontal plates of cast-iron, fitted with wrought-iron tubes, the joints between the ends of the tubes and the perforated plates being made by expanding the ends of the tubes in the usual manner.

The ammoniacal liquor enters the bottom chamber, *a*, through the tube, *b*, rises through the wrought-iron tubes to the middle chamber, *c*, then through the second set of wrought-iron tubes to the top chamber, *d*, whence it overflows along the pipe, *e*, to the top chamber of the still at a temperature approaching 200 Fahr. The waste gases from the saturator enter the pipe, *f*, after passing through a baffle box which arrests any acid spray mechanically carried forward. These baffle

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boxes usually consist of a short tube of large diameter, lined with lead, and in the cavity thus formed a number of circular

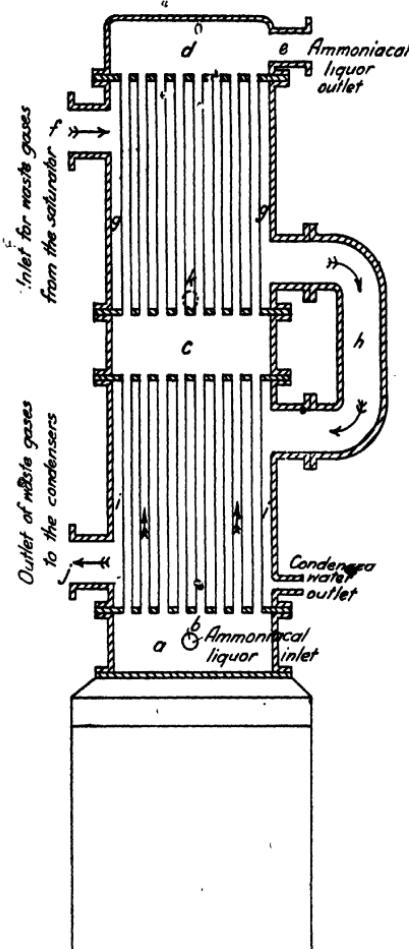


FIG. 3.—Vertical Superheater.

perforated lead discs are placed, against which the wet gases impinge. The condensings from the baffle box are led back

through a trapped tube to the saturator. After entering the superheater the gases play upon the outsides of the wrought-iron tubes in the chamber, *g*, then along the pipe, *h*, through the chamber, *i*, where they again play upon the outsides of the wrought-iron tubes and finally leave along the pipe, *j*, on their way to the condensing plant.

The liquid which condenses in the chambers, *g* and *i*, from the waste gases is of a very disagreeable nature and is known, somewhat appropriately, as "devil water." This is led away to a suitable seal pot, such as the one shown in Fig. 4, and then dealt with as described later on.

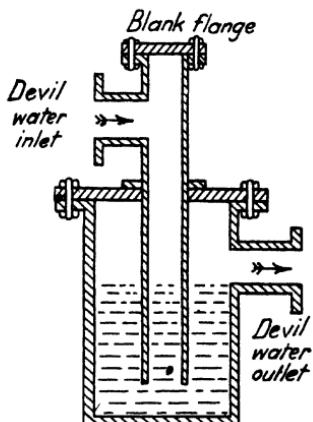
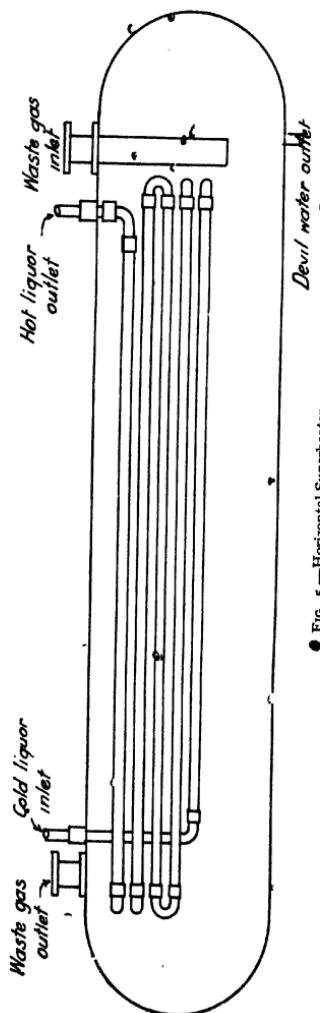


FIG. 4.—Seal Pot.

As there is a certain amount of expansion and contraction in starting up and stopping the plant, fears have been expressed that this would speedily lead to serious breakages in the wrought-iron tubes, but during nearly ten years' experience with them the writer experienced very little trouble through this cause. However, to meet the difficulty, Messrs Clapham Bros., Limited, introduced an improved form of superheater in which the bottom ends of the wrought-iron tubes are secured to the bottom perforated plate by a nut on the top side of the plate and another underneath the plate. The tops of the tubes pass through stuffing boxes, packed in the usual manner. This removes any fear of the tubes being injured by expansion or contraction.

In works where a fair amount of room is available for the sulphate of ammonia plant, an efficient superheater may be

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• FIG. 5.—Horizontal Superheater.

made from an old-fashioned egg-ended boiler or a disused steam boiler from which the tubes have been removed and the openings in the ends suitably blanked up. Inside, a coil is formed of

2-inch or 3-inch wrought-iron pipes, the connections being made with malleable iron double bends. Such an arrangement is shown in Fig. 5. In Figs. 6 and 7 are shown sketches of these double bends used in the works with which the writer was

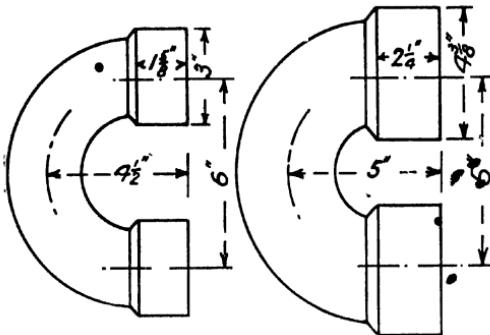


FIG. 6.—Malleable Iron Double Bends, for 2-inch W. I. Pipe.

FIG. 7.—Malleable Iron Double Bends, for 3-inch W. I. Pipe.

connected, and the sizes given are found to be very convenient ones. They are cheaper and more durable than wrought-iron bends.

In the pipe leading from the superheater to the still, it is desirable to place a socket for a thermometer, in order that one may be inserted, as the information furnished from this source is of considerable use in regulating the working.

## CHAPTER IV

### PLANT REQUIRED FOR THE MANUFACTURE OF SULPHATE OF AMMONIA (*continued*) : WALKER'S, COLSON'S AND DAVIS' STILLS

A GOOD typical circular still is shown in Figs. 8, 9 and 10 (pages 23, 24, 25). It is made by Messrs C. & W. Walker, Limited, and consists of a number of cast-iron sections, the joints somewhat resembling those of spigot and socket pipes, the whole being braced together by a number of long vertical bolts.

The joints are made of "Vulcan" (sometimes locally known as "Scotch") cement, liberally mixed with hemp gaskin (spun yarn) cut up into short lengths, the whole being thoroughly mixed up into a very stiff putty with boiled linseed oil. If this putty is carefully made it will be found to be a very effective jointing material, and steam may, if necessary, be turned on as soon as the joint is made. In cases where the joint blows it is usually found that the putty has been made too thin.

Underneath the nut at the top of each long vertical bolt is inserted a strong spiral spring. These allow for expansion and contraction in starting and stopping the plant, and if carefully adjusted they act as very effective safety valves, as if the pressure gets too high they would allow the top to lift a little and the joint to blow.

The ammoniacal liquor enters the still from the superheater along the pipe marked *a* in Fig. 8, fills the top chamber to the level of the overflow pipe marked *b*, flows down this into the second chamber and similarly through each succeeding chamber until, as waste liquor, it reaches the outlet in the bottom chamber of the still. By the time the liquor reaches the liming chamber, *c*, the bulk of the volatile ammonia has been driven off by the steam. Into this chamber the milk of lime is pumped through the pipe, *d*, and mixes intimately with the ammoniacal liquor in this and the succeeding lower chambers, and thus, with the steam, effects the liberation of almost the whole of the remaining ammonia.

The bulk of the steam enters the still through the pipe in the bottom chamber, marked *e*. Just after entering the chamber a tee is placed on the pipe and in each branch is screwed a semi-circular pipe perforated on the side facing the centre of the

chamber with a considerable number of small holes, thus dividing up the steam into a number of small jets. The steam passes

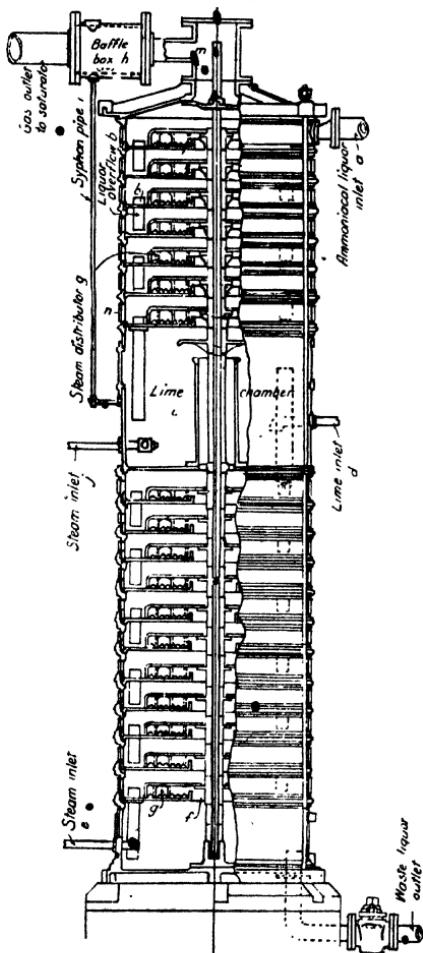


FIG. 8.—C. & W. Walker Ammoniacal Liquor Still.

upwards through the liquor in this bottom chamber (being prevented from passing away with the waste liquor by the

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intervention of a seal pot or other suitable device in the waste liquor pipe), gathers in the upper portion of the chamber, passes

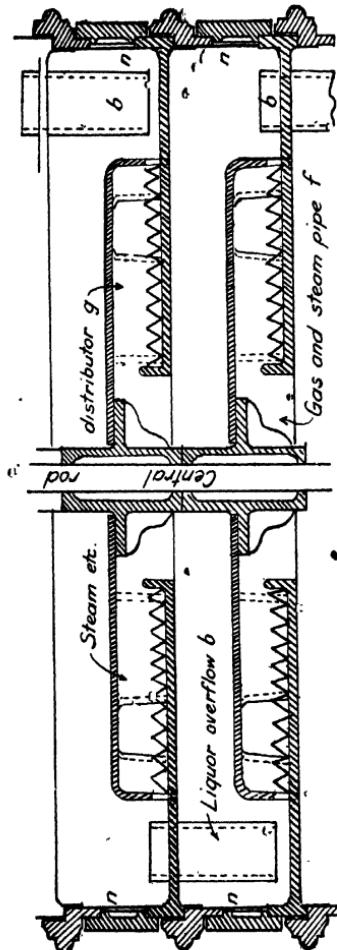


FIG. 9.—C. & W. Walker Sulphur Chambers.

through the oval opening, or pipe, *f*, in the bottom of the chamber above, and by means of the distributor, *g* (aptly called by the

workmen a "crocodile"), is divided into a very large number of small jets, which pass through the liquor just beneath its surface and carry with them the ammonia, etc. This is repeated chamber by chamber until the steam and gases issue from the top of the still through a baffle box, *h*, of similar construction to that previously described (page 17), to the saturator. The condensings and any spray carried forward mechanically are automatically returned to the still through the siphon pipe, *i*.

A second steam supply, *j*, similar in construction to that

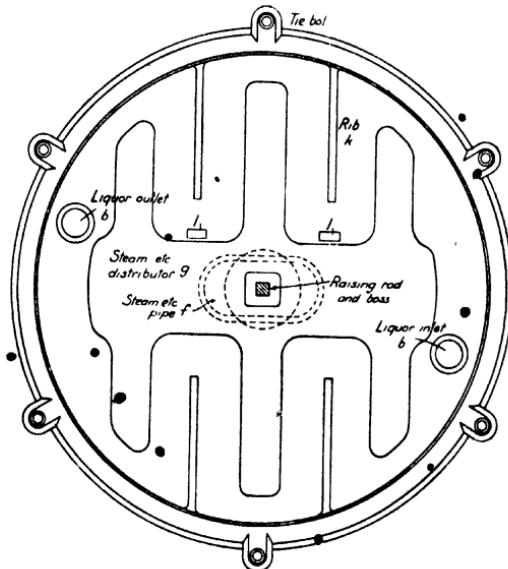


FIG. 10.—C. & W. Walker Still. Plan of a Chamber.

described above, enters the lower portion of the liming chamber, but the use of this is quite as much to agitate the contents of the chamber, and thus secure an intimate admixture of the milk of lime with the ammoniacal liquor, as to directly assist in the liberation of the ammonia.

Fig. 9 shows two chambers of the still drawn to a larger scale and Fig. 10 a plan of one of them. The references in these two plans are the same as in Fig. 8.

The ribs, *k*, shown in Fig. 10 deflect the steam upwards and prevent the jets from the teeth of the different portions of the distributor playing against each other, while the small blocks, *l*,

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assist in the setting of the distributors in position when erecting the still.

It will be noticed that the overflow pipes in the chambers

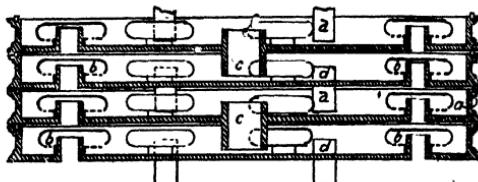


FIG. 11.—Colson Still. Section of Chambers.

are on alternate sides, thus obviously causing a longer flow of the liquor and consequently a more efficient contact with the steam.

A central rod passes through the still on which a number of

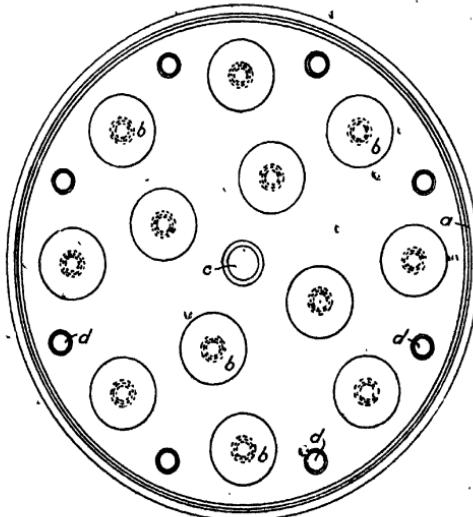


FIG. 12.—Colson Still. Plan of a Chamber

blocks and the steam distributors are threaded.\* By the removal of the top box, *m*, and the insertion of a screw arrangement provided by the makers, the rod and its appendages can be raised.

the idea being to provide a ready means of temporarily cleaning the still, but as it was not found to work very satisfactorily, in the later types of stills it has been superseded by much more efficient arrangements.

The still shown in these drawings is 18 feet  $2\frac{1}{2}$  inches high in the ironwork and 4 feet internal diameter.

About the time that this still was placed upon the market, several others were introduced by various makers. They differed from the Walker type chiefly in the method of joining

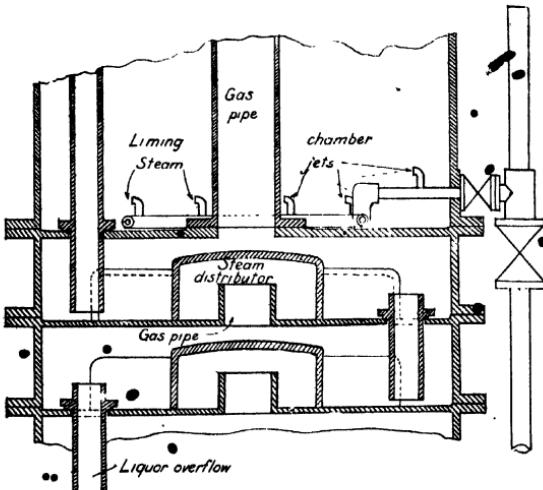


FIG. 13.—Davis Brothers Still. Vertical Section of Portions.

the sections, in the shape of the steam distributors, and in the arrangements of their overflow pipes.

In Figs. 11 and 12 (page 26) the still invented by the late Mr Alfred Colson, of the Leicester gasworks, and manufactured by Messrs Ashmore, Benson, Pease & Co., Limited, is shown.

As is the case with Messrs C. & W. Walker's still, this also consists of a number of cast-iron sections, but instead of a spigot and socket arrangement and a cementing material for the joints, in this case in the top of each section there is a square groove, *a*, in which fits a ring made of square india-rubber cord, standing above the surrounding metal. The joint is made by cutting the ends diagonally, and cementing them together with the solution used for repairing punctures in bicycle tyres. If the groove is painted with blacklead (graphite) made up with water,

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before the insertion of the india-rubber, it much facilitates the removal of the cord when this requires to be done. The whole still is bolted together with vertical bolts passing through lugs on the top and bottom sections, but these bolts are not provided with spiral springs, as is the case with Messrs C. & W. Walker's still, nor are they made long enough to receive them. The makers would no doubt be quite willing to make them longer and to provide the spiral springs if requested to do so, and the writer would strongly recommend this being done.

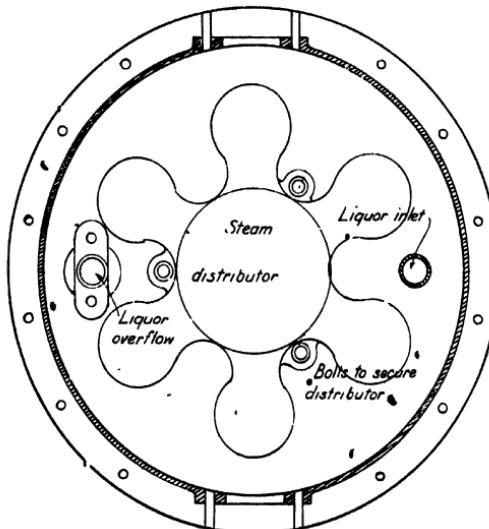


FIG. 14.—Davis Brothers Still. Plan of a Chamber.

The steam distributors, *b*, known as "roses," are shaped somewhat like a mushroom and have a ring of holes on their under surface to divide the steam into a large number of jets. They slide over the steam and gas pipes and a slight turn locks them by means of a "bayonet catch."

In one set of trays the liquor overflows, *c*, are larger and central in position, and in the alternating trays they are smaller and placed near the circumference of the still, *d*. By this means the liquor is very nicely distributed and very effective contact with the steam is secured.

In Figs. 13 and 14 (pages 27, 28) are shown details of the still supplied by Messrs Davis Brothers. In this still the sections

have planed flanges and are bolted together, the cementing material being either the familiar mixture of red and white lead or the "Vulcan" cement previously mentioned.

The steam distributors are of somewhat peculiar shape and are known as "spiders." Their teeth (not shown on the drawings) are square in shape. The distributors are bolted down to the trays as shown. The liquor overflows alternate in position as shown.

This still was practically the first column still to be introduced into this country and is made in sizes from 2 feet to 6 feet in diameter, making from five to forty tons of sulphate of ammonia per week from 10 oz. liquor.

Though the various types of stills described in this chapter have been at work for many years they are still working very efficiently, their chief drawback being that they are not so convenient to clean out as those of more recent type.

The length of time they will run without cleaning out depends chiefly upon the composition of the ammoniacal liquor used and the efficiency of its decarbonisation before entering the liming chamber. The writer's experience in Sheffield some years ago with stills made by Messrs C. & W. Walker, Limited, was that they ran about twelve or thirteen weeks between cleaning out and cleaning out again, the stills running continuously without stoppages during the whole of the time.

When the still requires cleaning out it is necessary to take it to pieces, and for this reason it is desirable, when first erecting the still, to make provision for this being done by leaving a space sufficiently large to pile the sections in column as close to the permanent site of the still as possible. Over the centres of the permanent site and the temporary one a steel H girder should be fixed sufficiently high above the top of the still to give ample allowance for the length of the blocks and sling chains used in lifting. This girder should be fitted with travelling blocks, a very suitable form of which is made by Messrs Herbert Morris, Limited. The sections are most easily lifted by a ring, to which is attached two chains, the outer end of each chain being terminated by a strong hook. In the case of Messrs C. & W. Walker's still these hooks are slipped into the two hand holes of the section. In the case of Mr Colson's still the ring is provided with three chains and the chain hooked round under the catches of the steam pipes.

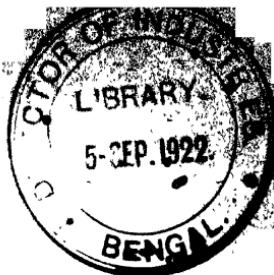
In taking a still to pieces for cleaning purposes, the top is lowered and run to one side, the sections are then lowered and placed upon each other in vertical column, and the distributor placed on one side, ready for cleaning. The bottom section will not require to be removed, as it can be cleaned *in situ*, but where the bottom ring and the bottom plate are not in one piece it is

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always desirable to re-make the joint. The sections are carefully cleaned, as are also the distributors and pipes, and they are then put together again. It is desirable to make one man responsible for the work; and he should see that everything is ~~cleaned~~ before the next tray is lowered, as it is obvious that the omission of anything will lead to endless trouble and expense afterwards.

In erecting a still it is necessary that each tray should be quite horizontal, as any deviation from this is a fruitful cause of difficulty in working the still, as it affects the seal and consequent flow of the liquor down the still.

In erecting a Messrs C. & W. Walker's still, the most convenient time to insert the central rod is just after the liming chamber has been placed in position.



## CHAPTER V

PLANT REQUIRED FOR THE MANUFACTURE OF SULPHATE OF AMMONIA (*continued*): RECENT TYPES OF STILLS—COLSON'S, WILTON'S, WALKER'S, COMBINATION, WYLD AND SHEPHERD'S

FROM our remarks in the preceding chapter it will be gathered that the chief objection to the earlier single column type of stills described therein was the difficulty connected with cleaning them out. As a rule the volatile or "free" portion of the still

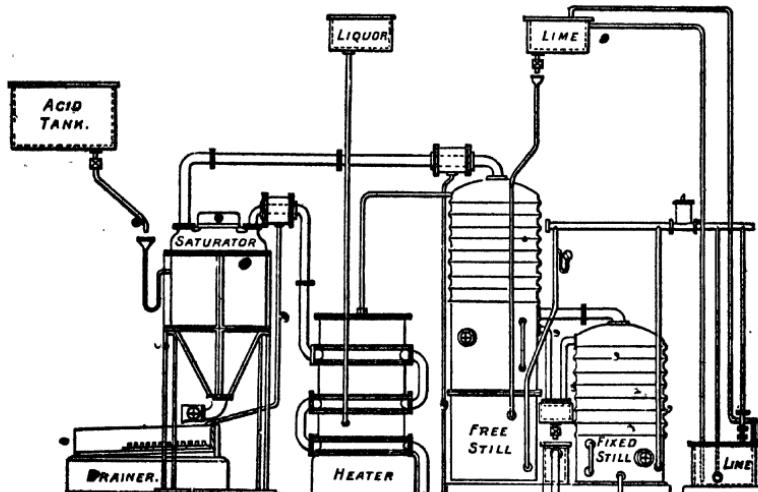


FIG. 15.—Colson Sulphate of Ammonia Plant.

would run for two or three years without cleaning out at all, the choke usually taking place in the liming chamber and the three or four chambers immediately below it. In the earlier single column stills, in order to clean out these choked chambers it is necessary to remove the whole of the volatile portion chamber by chamber before the defective portion can be reached, thus causing a considerable amount of unnecessary work. To overcome this difficulty the first important improvement was

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to erect the "volatile" chambers upon one base and the "fixed" chambers upon a second one, the liming chamber being placed at the base of the former series (in which case suitable manholes are provided, through which it may be cleaned *in situ*) or at the top of the latter series. Such an arrangement is shown in the still in Mr Colson's plant in Fig. 15 (page 31), and when room permits similar arrangements may be obtained in the various other makers' stills.

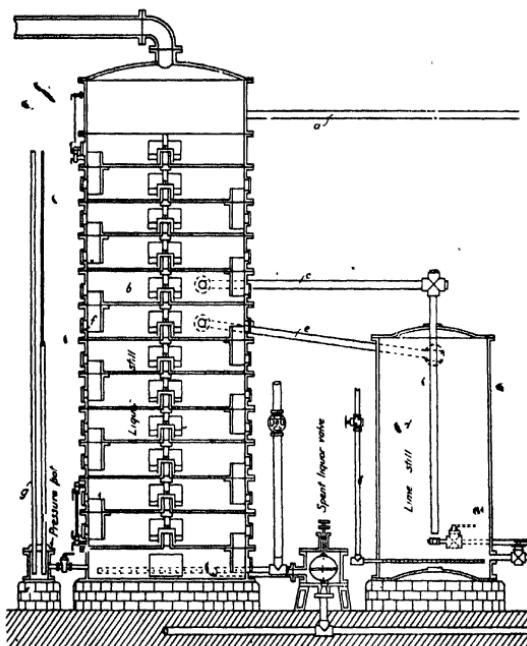


FIG. 16.—Wilton Still.

We will now proceed to consider two types of stills which, while they retain all the advantages of the earlier types, also provide most efficient methods for effectively cleaning them out with the whole of the chambers *in situ*.

The first of these is supplied by the Chemical Engineering and Wilton's Patent Furnace Company, Ltd., and is shown in Figs. 16 and 17. The ammoniacal liquor enters the still from the superheater, along the pipe, *a* (Fig. 16), and passes from chamber to chamber in the usual way until the one marked *b* is reached.

when, instead of passing through the overflow, *f*, the liquor descends through the pipe, *c*, by gravitation to the liming still, *a*, and passes back to the liquor still, mixed with milk of lime, along the pipe, *e*, and then descends in the usual course through the remainder of the still, leaving by the waste liquor valve, which will be described in the next chapter. The position of pipe, *c*, is rather lower than shown in our sketch, in order to compel the liquor to take that course in preference to rising and overflowing over the overflow pipe, *f*. At *g* is shown an efficient form of safety valve for an ammoniacal liquor still.

Fig. 17 shows the chief characteristics of this excellent type of still. The overflow pipes, *a*, are very large, and thus run much longer without making up, and when they do make up they are easily cleaned out after removing the lid which covers the opening, *b*. The man-lids are removed from the opening, *c*;

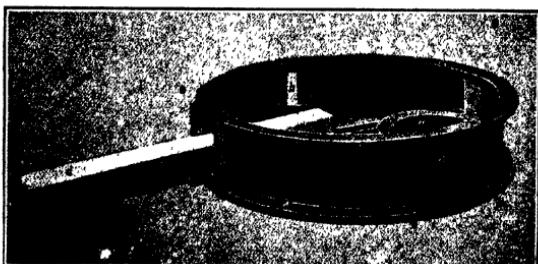


FIG. 17.—Wilton Patent Ammoniacal Liquor Still.

the distributor, *d*, taken out, cleaned, and, after the chamber is cleaned out through the openings, replaced; the lids are replaced in position, and the still is ready to resume work, the whole work of cleaning being done in a few hours. The drawings speak for themselves, and to those accustomed to stills the advantages are so obvious that no comment is necessary.

In the second type of still, made by Messrs C. & W. Walker, Limited, and shown in Figs. 18, 19 and 20, the same desirable ends are equally well accomplished by a somewhat different method. This still consists of square cast-iron sections, and the whole of the front and back plates, *a*, are removable, thus enabling the distributors, *b*, to be withdrawn, and the chambers cleaned *in situ*. The overflow pipes, *c*, are external, and as the outer portion consists of a removable flange, *d*, it is obvious that cleaning them out is an exceedingly simple process.

The chief objection to the employment of wrought-iron or steel boiler plates in the construction of stills has been that some

### 34. MANUFACTURE OF SULPHATE OF AMMONIA

constituent of the warm, raw ammoniacal liquor has had a very destructive effect upon these materials, but the writer's experi-

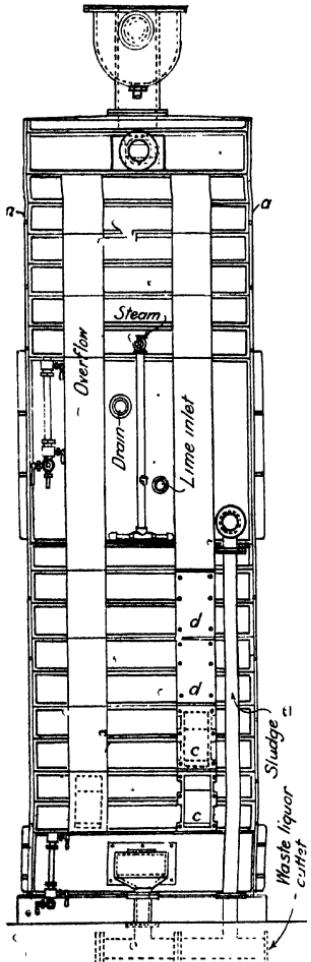


FIG. 18.—C. & W. Walker Still.

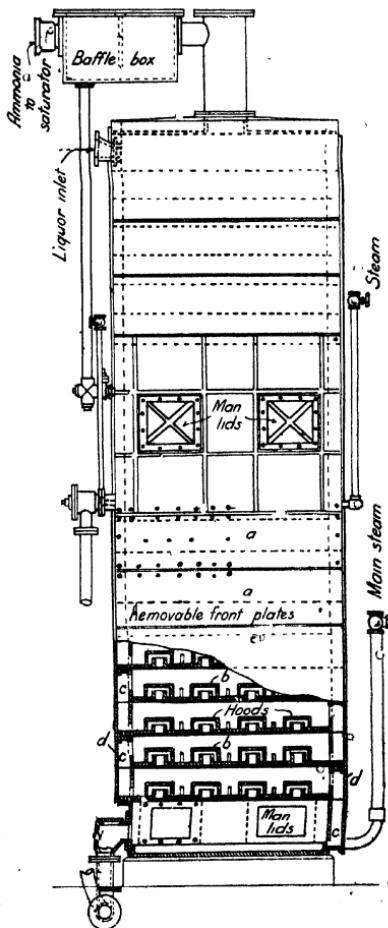


FIG. 19.—C. & W. Walker Still.

ence is that by the time the volatile gases are given off this injurious action is reduced to a negligible quantity. In the

works with which the writer was recently connected this knowledge was taken advantage of and a still (used, however, for a rather different process than sulphate of ammonia making), was designed and constructed, consisting as to the upper or "volatile" portion of a Colson's still, and as to the lower or "fixed" portion similar to a steam boiler. Such a still, modified to suit the purpose of sulphate of ammonia making, is shown in Figs. 21 and 22. The lowest chamber of the Colson's, or rather cast-iron, still requires to be somewhat modified by having a suitable flange on the bottom to bolt to the angle-iron riveted round the top of the wrought-steel section, and its liquor overflows, *a*, lengthened to be sealed in the contents of the liming chamber. The lower portion of the still is made like a steam boiler, and is divided into chambers by horizontal plates, either dished like a boiler end or they may be secured to the shell by means of angle-irons. The liming chamber and the bottom chamber are each 4 feet in height, the other chambers 1 foot 9 inches each. Manholes are provided on each side of the still through which the distributors are withdrawn and the men enter to clean out the still. If the bottom of the manhole is placed a little above the liquor level it assists materially in reducing the dirtiness of the cleaning operations, as by removing the plug, *c*, the liquid contents of the chamber run to the one below and finally escape through the waste-liquor pipe. The overflow and gas pipes are of light cast-iron, but wrought-iron pipes and flanges could be substituted with advantage. The distributors, *d*, are made of sheet-iron (a new departure), the corners being turned over and riveted, but the writer thinks a neater job would be made by using narrow angle-iron to rivet to. To prevent them overturning when at work there is a bar of flat iron under the gas pipe, through which a vertical bolt passes, and after passing through the top of the distributor it is secured by a nut, having two handles attached to it for convenience of manipulation. This arrangement is shown at *e*. If the still is well washed down by pumping

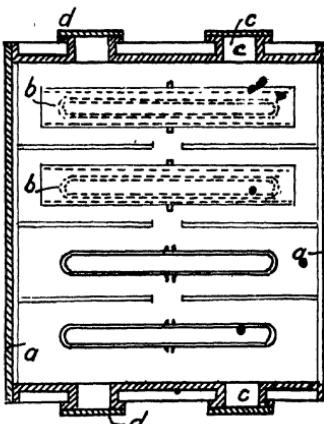


FIG. 20.—C. &amp; W. Walker Still.

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water down it, and the man lids removed, cleaning out is done without much difficulty.

An entirely new type of still construction is shown in Fig. 23. This shows the patent still introduced in 1907 by Messrs W. Wyld & Shepherd. (British patent, 8317/07.) This still consists of a number of square cast-iron sections forming the shell, but the interior of the still is not divided into chambers

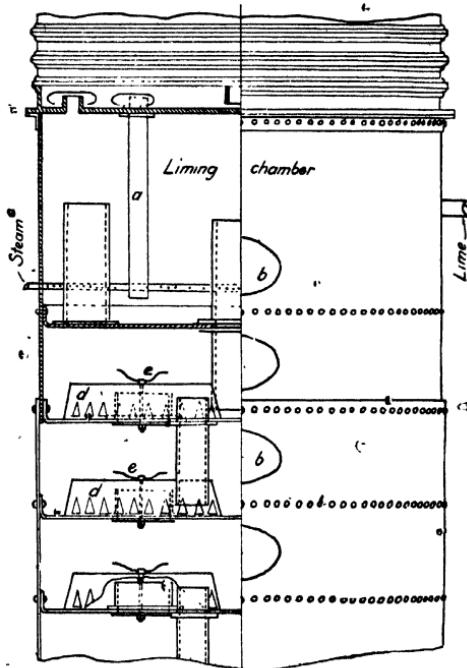


FIG. 21.—Combination Still—“Volatile” portion, cast-iron; “fixed” portion, steel boiler plates.

in the usual manner. Instead of this, there are small races, *a*, on the sides of the still, on which rest the very thin cast-iron plates, *b*. These are set at an angle and the distance apart regulated by the thickness of the small bosses, *c*, on the plates. Each row is placed at right angles to that below it. The liquor descends over these plates in a very thin film and, meets the steam and gases ascending through the interstices, thus ensuring very efficient contact. Lime is admitted in about the

usual position in the still. The principle is a very ingenious one, and the writer hears that this still is working in a very efficient manner.

A further new principle in still construction was introduced by Mr W. Wyld in 1911-12. (British patent, No. 25,716, 1910.) This still is shown in Figs. 24 and 25. In Fig. 24  $a$  is a circular still having its gas outlet at  $a^1$  controlled by the cock,  $a^2$ , so that the requisite pressure may be maintained in the still. After passing through suitable heaters, the ammoniacal liquor is forced in through the pipe,  $a^3$ , and by means of the centrifugal

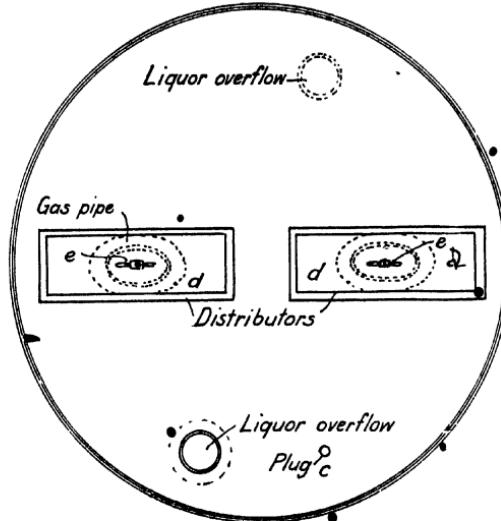


FIG. 22.—Combination Still. Plan of a Chamber.

spray nozzles,  $a^6$ , is discharged vertically in the form of a misty film into the still. Owing to the fine state of division the liquid undergoes when leaving the nozzles, and assisted by a little steam admitted through the perforated pipe,  $a^7$ , the gaseous constituents are immediately eliminated and pass out at  $a^1$ . To prevent the liquid leaving at  $a^1$  along with the gaseous products, baffle plates,  $a^8$ , or perforated diaphragms are arranged as shown.

From the still,  $a$ , the liquor overflows along the pipe,  $a^5$ , into the liming chamber,  $b$ . Milk of lime is pumped into this chamber through the pipe,  $b^1$ , and in order to keep it thoroughly agitated, a little steam may be passed through the perforated pipe,  $b^2$ .

The whole then overflows into the still, *c*, and is forced through the centrifugal spray nozzles by the pressure in the first still. In this still also there is a perforated steam pipe, *c*<sup>1</sup>, to assist the distillation. The waste liquor is finally pumped away through the pipe, *c*<sup>2</sup>.

In order to avoid putting so much pressure upon the still, *a*, vacuum may be employed at the exit of the system in which case the centre of the system would be worked at approximately atmospheric pressure.

Instead of the single still, *a*, at the beginning of the system, a series of two, three or more may be used, as shown in Fig. 25. A perforated steam pipe (not shown in the drawing) is placed in each still.

The writer understands that this still has been at work for some time, giving very satisfactory results. In the event

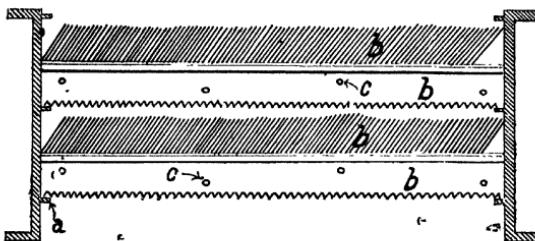


FIG. 23.—Wyld & Shepherd's Still.

of the choking up of a nozzle, provision is made whereby it can readily be removed and cleaned, but little trouble is caused through this occurring.

Yet another principle in still construction is introduced by Mr G. Keville Davis in his patent "Trepex" ammonia still, shown in Figs. 26 and 27. The principle upon which this apparatus works is based upon creating a spray of the liquid to be distilled by mechanical means and passing the steam and vapour through this spray. The ordinary column still contains a number of sprays down which the liquid travels, and through the liquor lying on them the steam and vapour is forced to double. The mechanical spray provides a much larger area of contact between the liquid on the one hand and the gas on the other.

Referring to the illustration it will be seen that one form of the apparatus takes the shape of a square cast-iron chamber divided by means of through partitions into compartments corresponding to the trays of the column still. Though this chamber are provided two longitudinal shafts which carry

fanners, marked *F*, and water lifting wheels, marked *W*. The latter work inside a guard, marked *G*, the purpose of which will be explained later. There are thus one fanner and one water wheel in each compartment. The first sprays up the liquid lying on the bottom of the apparatus, and this is just

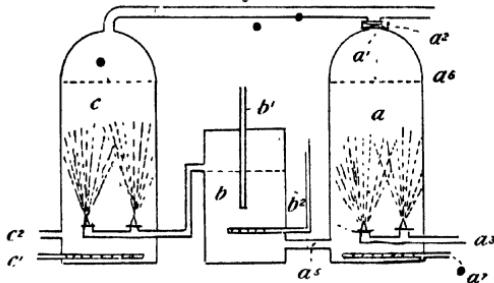


FIG. 24.—Wyld's Still.

level with the tips of the blades at their lowest point. The rapid rotation of the fanners causes a very fine spray to be made and thus exposes a very large surface to the action of the steam. The gas or vapour passes from chamber to chamber through passages which are concentric with the fanners, and the position of the fanners is such that the tendency is by their

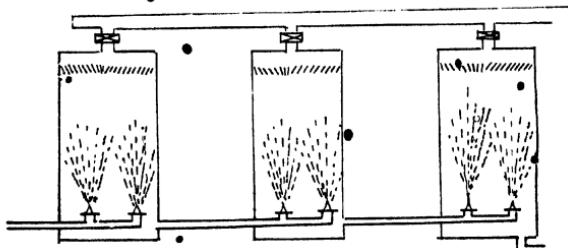


FIG. 25.—Wyld's Still.

rotation to draw the gases from one chamber to the next. It has been found that there is occasionally difficulty in getting the liquor to flow through such an apparatus, and for this purpose the water wheels are provided. They lift the liquor and throw it against the specially shaped guard, *G*, and into the next chamber to the one in which the fanner works. The flow

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of liquor is therefore made absolutely positive and there is no danger of choking up of any kind.

The first apparatus has now been at work upwards of eighteen

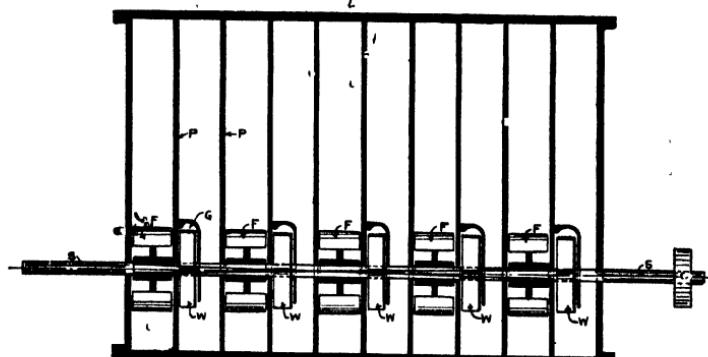


FIG. 26.—Davis' "Trepex" Still.

months and has proved itself a more efficient apparatus than the old column still, besides being easier to work and requiring less attention.

In the case of small works where the quantity of ammoniacal liquor or the labour available do not warrant the installation of

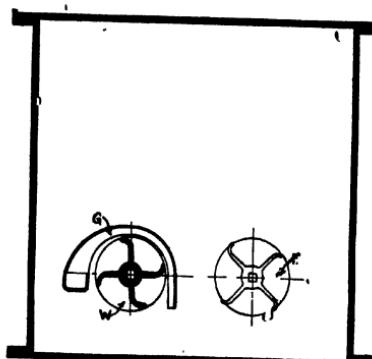


FIG. 27.—Davis' "Trepex" Still.

a continuous plant, Messrs C. & W. Walker, Limited, supply a very neat little still shown in Figs. 111 and 112, Chapter XIV., which does not encroach upon the steam supply of the works as it is heated by a coke or coal fire.

## CHAPTER VI

### PLANT REQUIRED FOR THE MANUFACTURE OF SULPHATE OF AMMONIA (*continued*) : LIMING APPARATUS

FROM our previous remarks it will be seen that in order to liberate the "fixed" ammonias from the ammonical liquor it is necessary to add a quantity of lime in the form known as "milk of lime". We will now describe the apparatus necessary for dissolving the lime and introducing it into the still. The lime may be slaked by (a) waste liquor from the still; (b) hot water, or (c) cold water. Of these methods the writer prefers the first-named, as by its use, should the contents of the still run short of lime at any time, on the spent (or waste) liquor entering the lime-dissolving tank and coming in contact with the lime there, the smell of ammonia is so evident that it immediately warns the men in charge of the process of the condition of affairs, and enables them to take steps to promptly rectify it.

It is scarcely necessary to point out that if the waste liquor is used for this purpose it should be taken off at a point where either the pressure or height gives sufficient force to supply the liming plant. The best places to obtain the supply are (1) the bottom chamber of the still; (2) the pipe leading from this chamber to the seal pot, or (3) from the seal pot itself.

Methods (a) and (b) are preferable to method (c), as it is obvious that the use of hot milk of lime means a considerable saving of fuel compared with the cold method. In cases where methods *a* and *b* are adopted it is desirable to carry away the steam given off by means of hoods and wooden chimneys of the usual pattern.

A typical liming apparatus is shown in Fig. 28 (page 42), this being the one supplied by Messrs C. & W. Walker, Limited, with their sulphate of ammonia plants. The lime is slaked in the tank, *A*, and run through the tap through the screen, *B*, into the tank, *C*, whence it is pumped by the single ram pump, *D*, into the liming chamber of the still. The pump also works the agitating arrangement, *E*, to prevent the lime settling down, and thus secures regular strength of the milk of lime and consequently steady working. The screen is covered with wire gauze about  $\frac{1}{8}$  in. to  $\frac{1}{16}$  in. mesh; and care must be taken

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to maintain this in good condition, as any holes allow small bits of lime to go through, and these give endless trouble in the

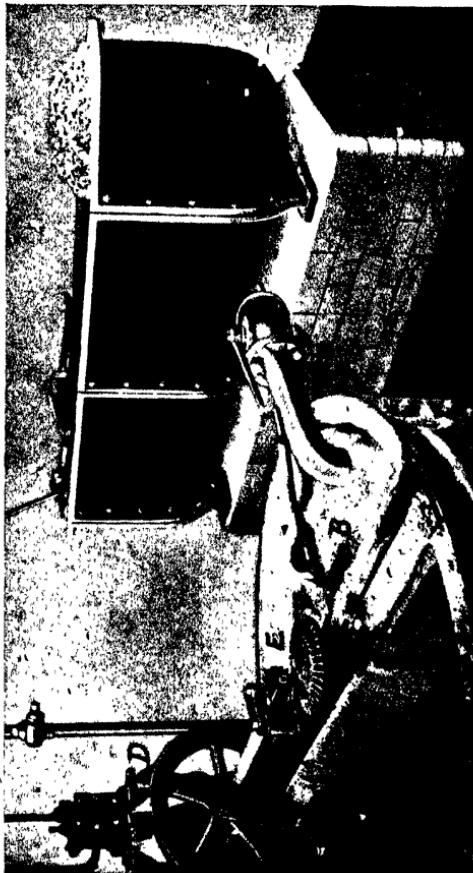


FIG. 28.—Lining Apparatus.

clacks of the pump, thus leading to the still running short of lime and causing "high" spent liquor samples.

Where a driving shaft is available, the lime-mixing arrangement shown in Fig. 29 (page 43) is found to be a very efficient one. The waste liquor is run into the circular tank, *a*, and the lime placed gradually on the perforated plates, *b*, the dissolving

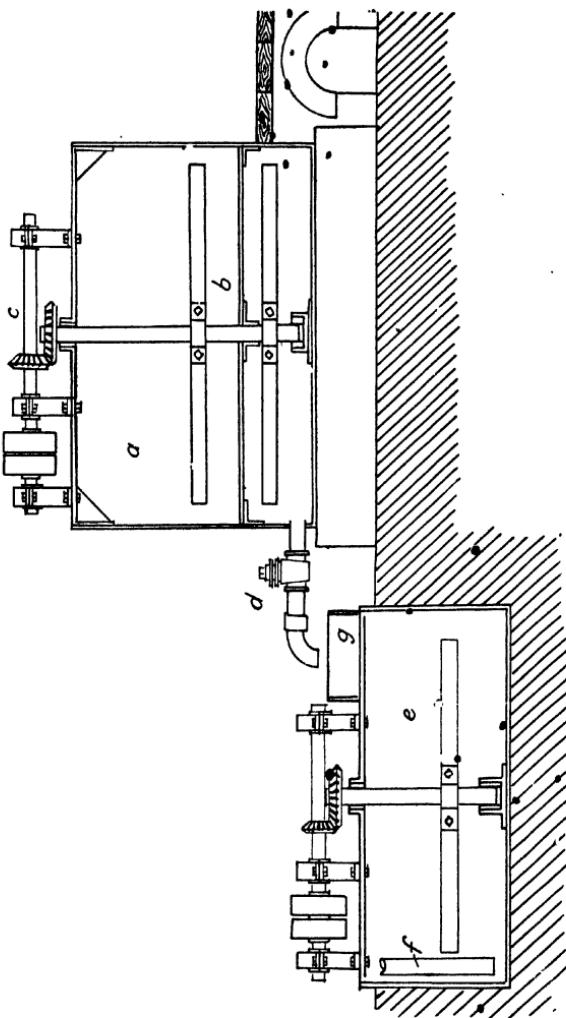


FIG. 29.—Lime Mixers.

being accelerated by the mechanical stirring of the apparatus, *c*. The undissolved stone is removed from time to time; and if its relative weight compared with the weight of lime used be

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FIG. 30.—Kestner Elevator.

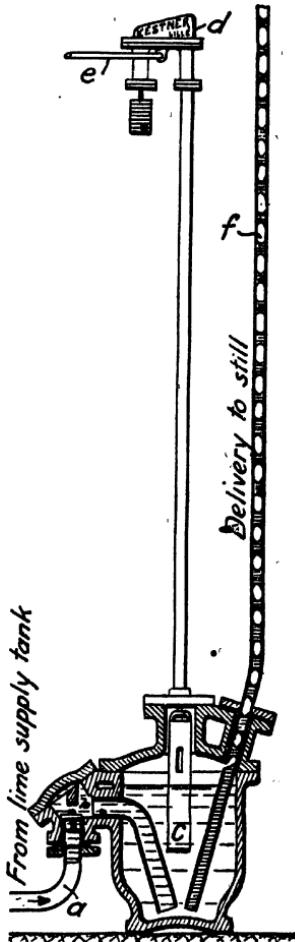


FIG. 31.—Kestner Elevator.

ascertained it forms a good guide as to the quality of the lime, as mentioned in Chapter II. The milk of lime runs through

## PLANT FOR MANUFACTURE OF SULPHATE

the cock, *d*, and through the screen, *g*, into the tank, *e*, whence it is pumped through the suction pipe, *f*, to the liming chamber of the still. In the tank, *e*, the contents are kept agitated by a similar arrangement to that in tank, *a*.

In works where there is a supply of compressed air available

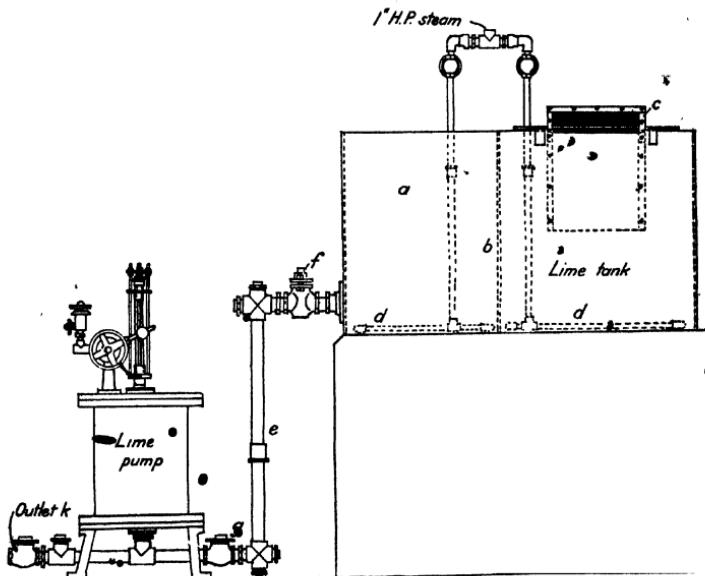


FIG. 32.—Wilton Patent Liming Apparatus. Elevation.

the lime pump may be replaced by an elevator such as is usually used for the elevation of sulphuric acid.

Such an arrangement is shown in elevation in Fig. 30 and in sectional elevation in Fig. 31. The milk of lime enters the automatic elevator from the supply tank by gravitation through the pipe marked *a* in Fig. 31, the supply being controlled by a cast-iron cock placed in this pipe. Its return is prevented by the valve, *b*. As the elevator fills, the float, *c*, gradually rises and by means of a rod attached to its upper end, when the elevator is full, it opens a valve in *d*, which admits the compressed air from the pipe, *e*, which forces the milk of lime up the pipe, *f*, to the liming chamber of the still. As the elevator

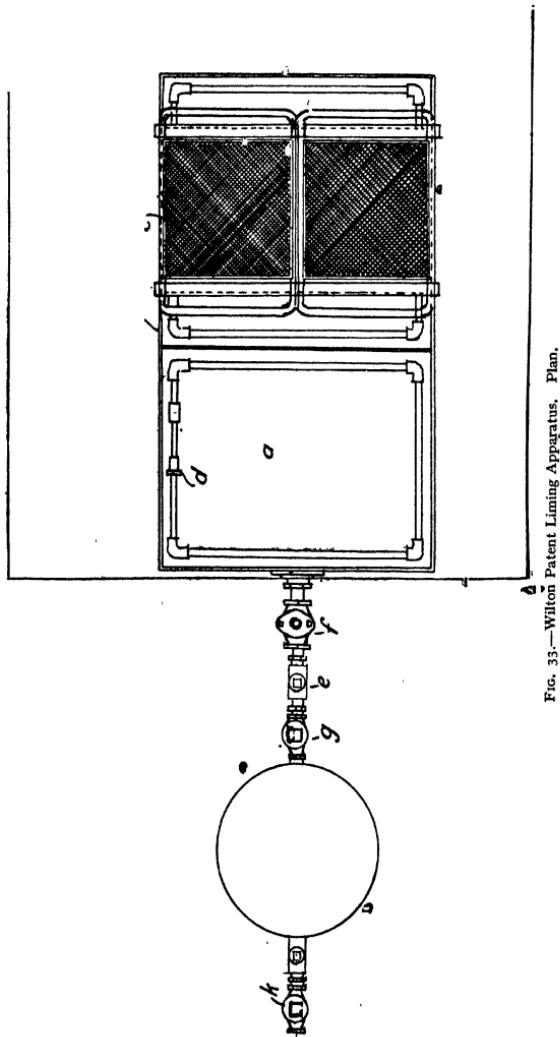


FIG. 33.—Wilton Patent Liming Apparatus, Plan.

empties the float gradually falls, and when the elevator is empty the rod closes a valve on the air supply and opens a valve in *d*.

which releases the pressure and forms a vent to allow the elevator to refill. In fixing these elevators it is desirable to have the pipe, *f*, as nearly vertical as possible to its full height, in order to prevent "air-locks." If a quadrant is placed on the supply cock in pipe *a* it will assist materially in the regulation of the supply.

A very efficient complete patent liming arrangement is

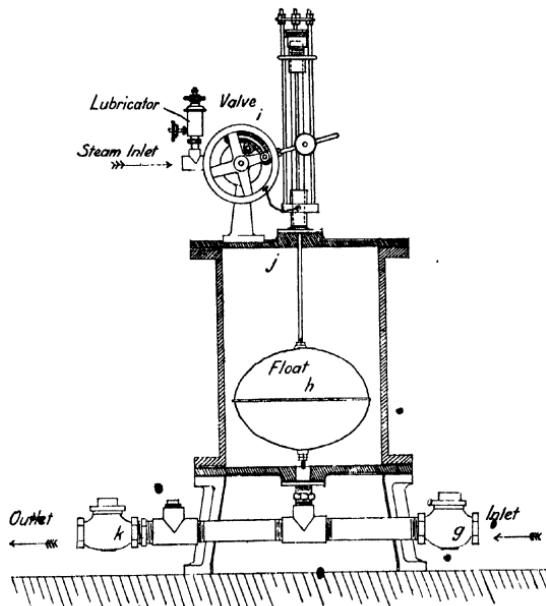


FIG. 34.—Wilton Patent Lime Pump.

made by the Chemical Engineering and Wilton's Patent Furnace Company, Ltd., and is shown in Figs. 32, 33 and 34. The lime slaking pan, *a* (Figs. 32 and 33), is provided with a division wall, *b*, and wire cages, *c*. The solid lime is put into these cages and is slaked by means of some pent liquor obtained as previously mentioned. The solution is agitated by the steam jets, *d*. The heavy sand from the lime remains behind the division wall, and is blown off about once a day, or when necessary. The milk of lime passes over the division plate, and enters the pump by

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gravitation through the pipe, *e*, the speed being controlled by the cock, *f*, and its return to the tank, *a*, being prevented by the check valve, *g*. As the pump fills, the float, *h* (Fig. 30), rises, and when the pump is full the rotary valve, *i*, is thrown over and admits the steam to the body of the pump at *j*. The steam blows the lime out of the pump body past the check valve, *k*, and thence into the plant. As the pump empties, the float drops and finally pulls back the rotary valve, *i*, which shuts

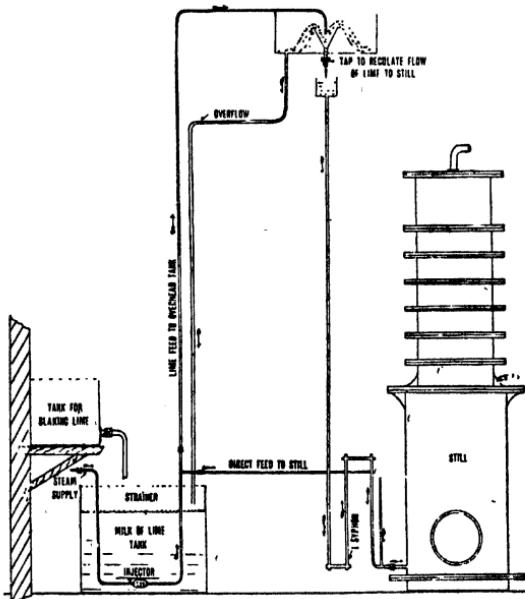


FIG. 35.—Shewring's Liming Apparatus.

off steam and opens the exhaust, allowing the pump to again fill with the milk of lime. The advantages claimed for this pump are :

- (1) The steam pressure does not affect the speed of the pump.
- (2) The pump when once adjusted continues to work at absolutely the same constant rate.
- (3) The speed is governed by the cock, *f*, on the lime inlet and not by the steam valve, as is the case with ram pumps.

## PLANT FOR MANUFACTURE OF SULPHATE 49

(4) The body of the pump is not machined, and there are no working parts in contact with the lime, therefore

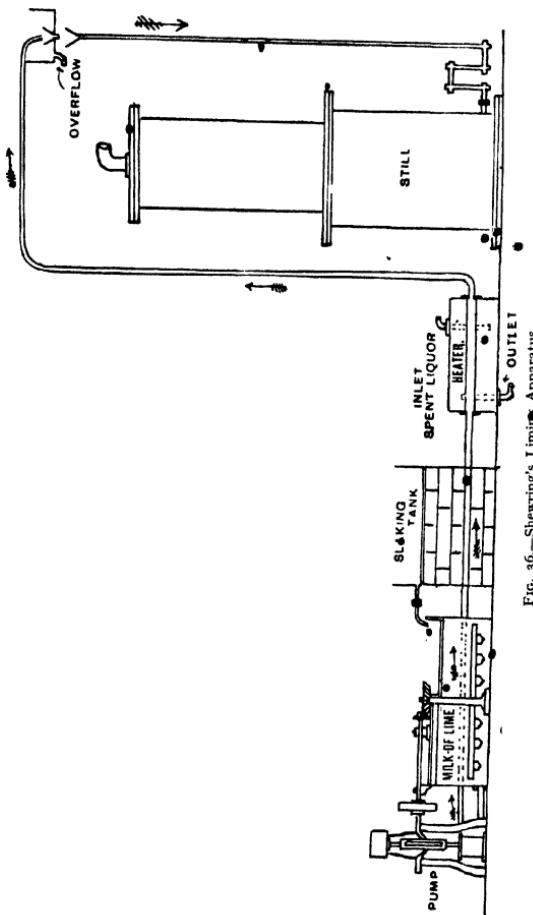


FIG. 36.—Shrewding's Lime Apparatus.

boiling milk of lime can be used without corrosive effect on the pump.

In some illustrations of small plants a hand pump is shown to pump the milk of lime into the still. This arrangement the

## 50 MANUFACTURE OF SULPHATE OF AMMONIA

writer would deprecate, as it is bound to lead to irregularity of supply. It is obvious that at the time of pumping there is likely to be a large excess (and consequent waste) of lime, while

as the time approaches for a fresh charge there will be a scarcity, and consequently "high" spent liquor.

It cannot be too strongly impressed upon the workmen that not only is it necessary that a certain quantity of lime should be put into the still in the twenty-four hours, but that it should be put in steadily during the *whole* of the time.

Another drawback to the intermittent supply is that if the milk of lime is colder than the contents of the still, it leads to a sudden cooling of the latter, a quicker rush of liquor through the still, and insufficient steam to secure effective distillation.

To those whose experiences, like the writer's, has been confined to large works where the above facts have been looked upon as the most elementary truths of sulphate of ammonia manufacture, it comes as a great surprise to learn that there are many works (chiefly of the smaller kind) where the importance of the steady supply has not been realised, and the writ

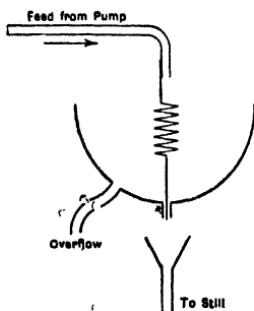


FIG. 37.—Shewring's Liming Feed.

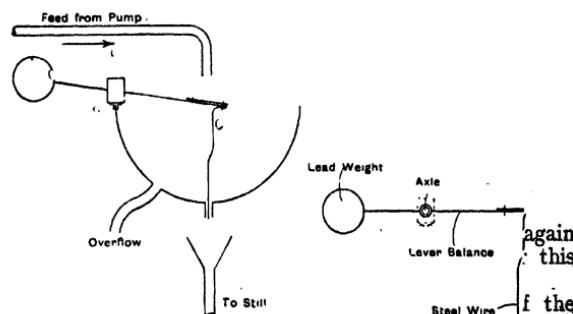


FIG. 38.—Another Shewring Lime Feed.

ork at

thinks that the thanks of the community are due the inlet Shewring for the manner in which, through the pages of the GAS WORLD, he has striven to drive home these facts.

In cases where a pump is employed it should be driven by either steam or other power, and if when going at its slowest speed it still pumps more milk of lime than is required, a small by-pass pipe and tap can be put on the delivery pipe of the pump, and arranged to return the excess to the supply tank.

A very simple arrangement to meet such cases is suggested

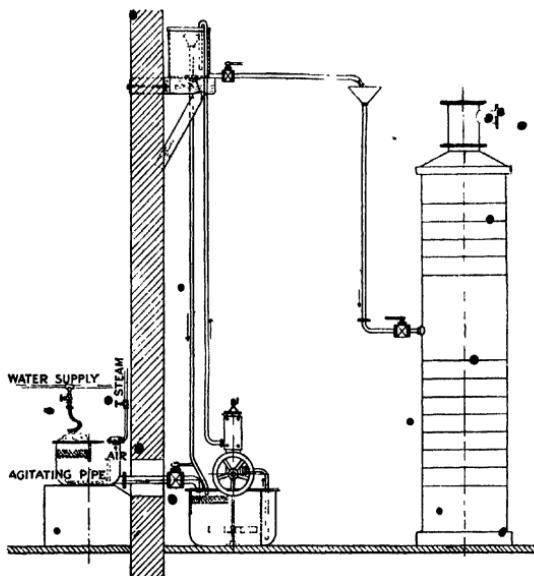


FIG. 39.—C. & W. Walker's Continuous Lime Feed.

by Mr Shewring in THE GAS WORLD of 19th October 1912, and is shown in Fig. 35.

Another arrangement is shown by Mr Shewring in an article in the same journal of 21st December 1912, now reproduced in Fig. 36.

These figures show the arrangements so clearly that no further explanation is necessary.

To avoid any chance of the pipe leading to the delivery pipe to the still choking up, Mr Shewring has devised the two simple but ingenious pieces of apparatus shown in Figs. 37 and 38. But a very little attention by the man in charge, in

## 52 MANUFACTURE OF SULPHATE OF AMMONIA

probing through the short pipe occasionally, should remove any difficulty in this direction.

Messrs W. C. Holmes & Co., Limited, are makers of Mr Shewring's continuous liming devices.

Messrs C. & W. Walker, Limited, also supply a continuous liming apparatus, embodying somewhat similar principles, and this is shown in Fig. 39.



## CHAPTER VII

### PLANT REQUIRED FOR THE MANUFACTURE OF SULPHATE OF AMMONIA (continued) : WASTE LIQUOR VALVES, COOLING ARRANGEMENTS, ETC.

ON the waste liquor outlet pipe from the bottom of the still a cock is provided to regulate its flow, and to prevent the escape of any live steam with it a suitable seal pot is also inserted in the pipe run. Such pots are shown in Figs. 40 and 41 (pages 54, 55), the former being sunk in the ground and the latter stood upon it in a convenient position near the still. If the seal pot is inside a building or other place where the escape of steam is objectionable, a cover may be provided with a vent pipe to lead the steam away, as shown in Fig. 40, but where the escape of steam is not detrimental, an open pot, such as is shown in Fig. 41, assists in the cooling of the spent liquor.

Instead of a seal pot such as we have just described, the spent liquor is occasionally led through a boiler arranged similarly to the superheater shown in Fig. 5, the heat being used to heat either water or ammoniacal liquor passing through the coil of pipes, while the requisite seal is obtained by regulating the height of the waste liquor outlet pipe. This method, however, cannot be highly recommended, as the lime sludge quickly deposits upon the pipes and renders them ineffective, and the necessary cleaning is then a matter of considerable difficulty.

A marked improvement upon the seal pot arrangement is the substitution of a special valve, such as that shown in Fig. 42 (page 56). This valve is supplied by the Chemical Engineering and Wilton's Patent Furnace Co., Ltd. *a* is the branch attaching the valve to the liquor still, and *b* the branch leading to pipe or trough conveying the spent liquor to the cooling tank. The object of the valve is to release the spent liquor from the still without loss of steam. The valve will perform this work with any pressure on the inlet. As the liquor passes into the valve at *a* the float, *c*, rises and draws up the plug, allowing the liquor to pass out. Immediately the liquor has left the valve and steam enters, the float drops back and the operation is repeated. In practice the float automatically takes up a position which allows the spent liquor to flow freely away as soon as it arrives at the bottom of the still.

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It thus removes the necessity for the constant supervision of the man in charge of the still, and does away with the frequent trouble caused by the choking of the long seal pot.

From the seal pot or automatic valve the spent liquor is

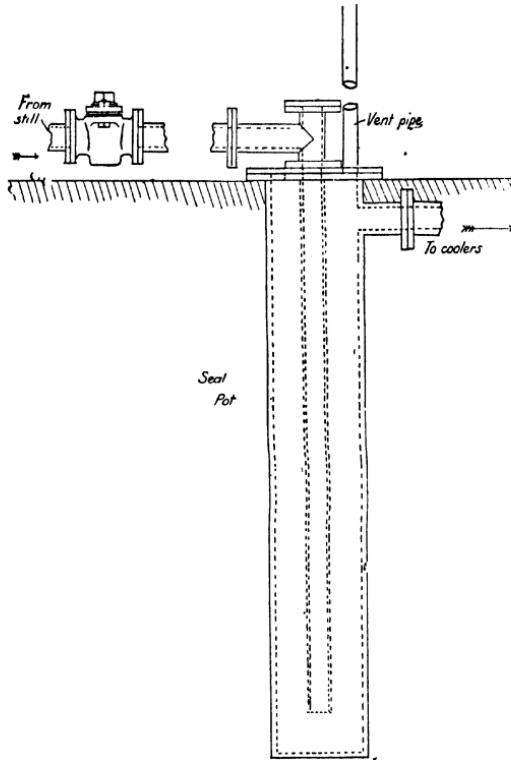


FIG. 40.—Seal Pot.

conveyed along flanged pipes or an open trough of cast-iron, wood, or bricks laid in cement to the settling and cooling tank. Where the escape of steam is not objectionable, the open trough is preferable, as it not only assists the cooling, but it is also so easily cleaned out. Where pipes are used they will require taking up frequently for clearing purposes, so the advantage of flanged over socket pipes for this purpose is obvious.

The requirements of the different local authorities differ somewhat as to the condition in which they desire the spent liquor to enter the sewers, but a little friendly consultation will generally lead to an arrangement that will be agreeable to both parties. In one city known to the writer, where the general sewage is of a slightly acid character (through metal

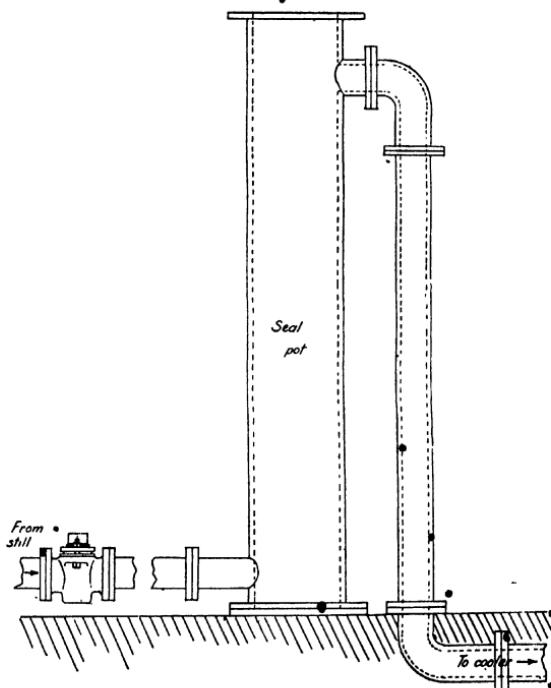


FIG. 41.—Seal Pot.

washings), the authorities allow the whole of the lime sludge to go into the main sewer along with the waste liquor, as it helps to neutralise the acid in the general sewage and renders it more suitable for bacteriological treatment, and they only stipulate that the temperature shall not exceed  $40^{\circ}$  Fahr. as it enters the sewer. If it exceeds this temperature it causes considerable inconvenience to the men working in the sewers. In this case the spent liquor enters one of the very large main

## 56 MANUFACTURE OF SULPHATE OF AMMONIA

sewers of the city, through which there is a large quantity of sewage passing, so that the chance of any inconvenience being caused by deposition of lime is a very remote one. In such an exceptional case as this a cooling tank, such as is shown in Fig. 43, is suitable.

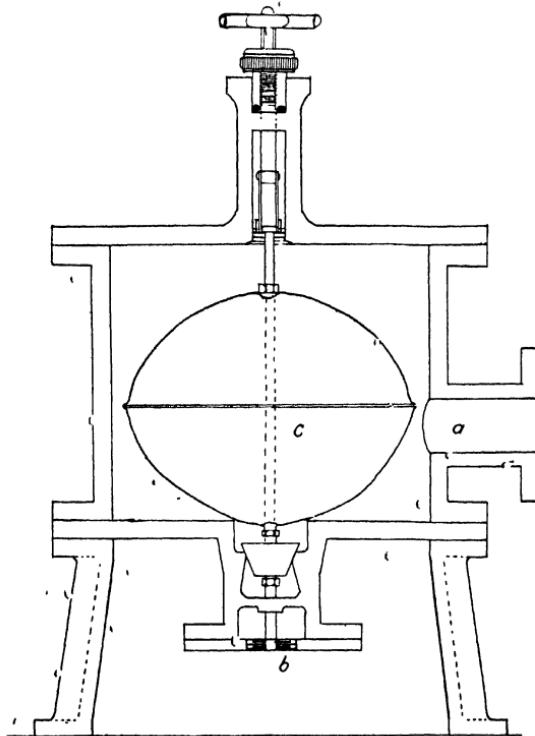


FIG. 42.—Wilton Patent Spent Liquor Valve.

The tank has a concrete floor and the walls are of brick-work laid in cement and sand. The pipes in the tank are of 3-inch wrought-iron, and as they have to be frequently taken out for cleaning purposes, the desirability of having so many flanged joints is at once obvious. In this case the spent liquor runs through the pipes, while cold water is run into the tank at the point shown. The slightly-warmed water overflows at

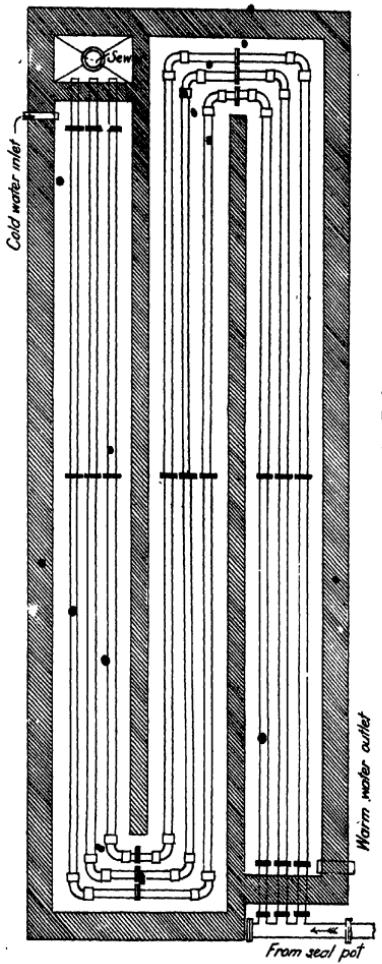


FIG. 43.—Spent Liquor Cooling Tank.

a point also shown, and if sufficiently pure in quality may be run to a suitable receptacle and then used for feeding steam boilers of other purposes.

Where, however, the sewer that the spent liquor enters is

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of but small area, or there is but a slow current through it, it is obvious that the lime sludge would deposit, block it, and cause very serious trouble. In such cases some means must be adopted

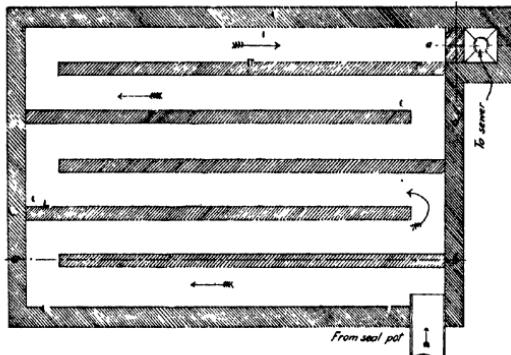


FIG. 44.—Tank for Settling and Cooling Spent Liquor. Plan.

to not only cool the spent liquor, but also to retain the bulk of the lime sludge. An arrangement suitable for this purpose is shown in Figs. 44, 45 and 46. This tank has a concrete floor, and the brickwork walls are laid in Portland cement and sand.

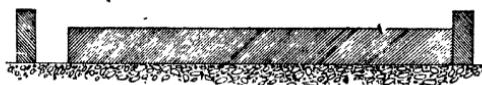


FIG. 45.—Sectional Elevation on Line a-b.

The spent liquor flows through the channels and is aerially cooled, the lime depositing along the channels and being retained by a weir near the sewer. The lime sludge is cleaned out from time to time, as required.



FIG. 46.—Sectional Elevation on Line c-d. Sectional Elevation on Line e-f.

## CHAPTER VIII

PLANT REQUIRED FOR THE MANUFACTURE OF SULPHATE OF AMMONIA (continued): SATURATORS OF VARIOUS TYPES—HAND-FISHED, EJECTOR DISCHARGED, BOTTOM VALVE DISCHARGED

AFTER leaving the baffle box described in Chapter IV, the ammonia, steam and other gases from the still enter the saturator. This important piece of plant receives its name from the fact

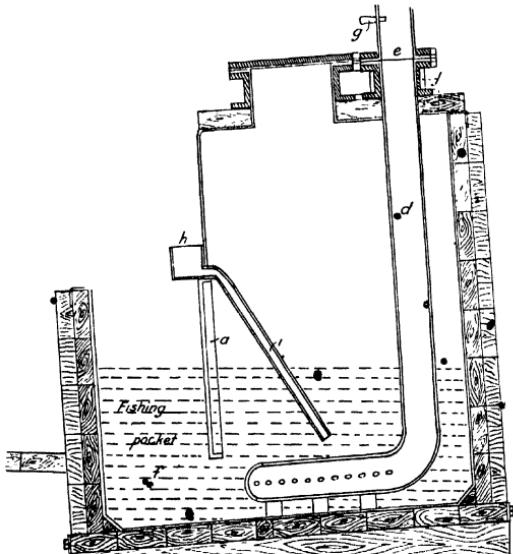


FIG. 47.—Hand-fished Saturator. Side Sectional Elevation.

that here the sulphuric acid is saturated with ammonia gas in order to form the sulphate of ammonia. A saturator is also sometimes locally known as a "blower," from the fact that the ammonia here blows into the sulphuric acid.

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According to the method adopted for the removal of the sulphate of ammonia formed, saturators may be divided into three types :—

- (a) Those from which the sulphate of ammonia is removed by " hand fishing."
- (b) Those from which it is removed by means of a steam ejector, and
- (c) Those from which it is removed by means of a suitable bottom discharge valve.

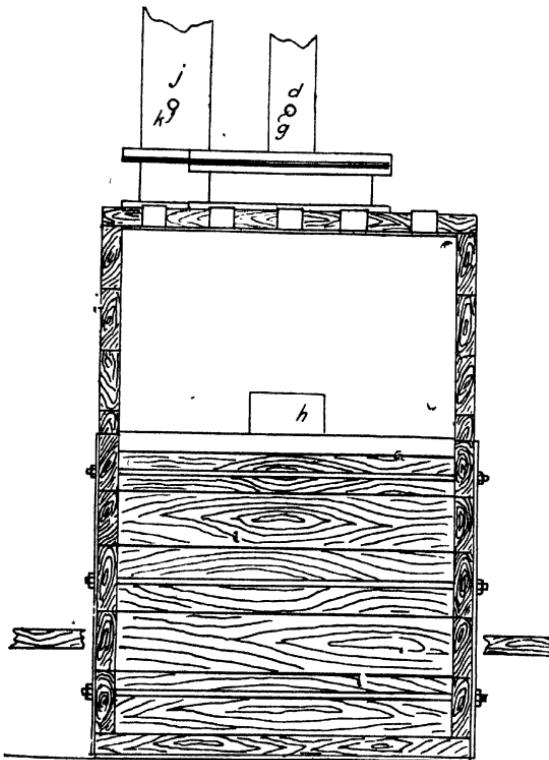


FIG. 48.—Hand-fished Saturator. Front Elevation.

Again, they may be further divided into "open" or "closed" types, according to whether much or little of the liquid contents of the saturator are exposed to the air.

The hand-fished saturators of type (a) are necessarily of the open type, those of type (b) may be either of the open or closed kind, and those of type (c) are generally of the closed character.

A simple type of open saturator suitable for hand fishing is shown in Figs. 47, 48 and 49. It consists of a wooden tank formed of 9-inch  $\times$  3-inch red-wood, securely bolted together and lined with sheet lead 20 lbs. per square foot. The midfeather, *a*, is of cast lead about  $\frac{1}{2}$  inch in thickness, stiffened with border, *b*, and ribs, *c*, an extra one inch thick, as shown in Fig. 49. The ammonia, etc., enters the saturator through the

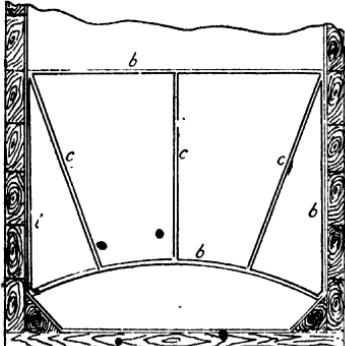


FIG. 49.—Hand-fished Saturator. Back of Midfeather.

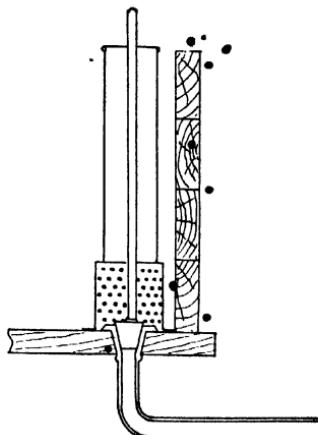


FIG. 50.—Plug and Seat for Acid Tank.

pipe marked *d*. From the flange joint, *e*, this is made of drawn lead pipe from 4 inches to 6 inches in diameter, according to the size of the saturator. This pipe should be of the best soft chemical lead, as should all the lead used about a sulphate of ammonia plant. The end nearest the fishing pocket, *f*, is bossed up by the plumber, and a lead flange of suitable size is cast and "burned" on the top end. A number of  $\frac{1}{4}$ -inch holes are drilled in the pipe in either one or two rows, each hole being drilled at an angle so that the ammonia blows towards the fishing pocket. In the iron pipe which brings the gas to this lead one a  $\frac{1}{4}$ -inch hole is drilled, and this is closed by the tapering wooden plug marked *g*. This plug is removed when the still is cooling down after stopping the plant, to prevent the vacuum sucking back the contents of the saturator.

## 62 MANUFACTURE OF SULPHATE OF AMMONIA

The acid and mother liquor are supplied from two overhead tanks along lead pipes, the flow being controlled by either earthenware taps covered with lead or regulus taps discharging into the small box, *h*. Provision should be made for shutting off the supply to these pipes at the tank end, either by regulus plugs and seats, as shown in Fig. 50, or by syphon box, as in Figs. 51 and 52. When the plugs and seats arrive the iron shaft in the plug will be found to be about 15 inches long, and this will require to be lengthened by welding on an iron rod of the requisite dimensions. The whole of the iron

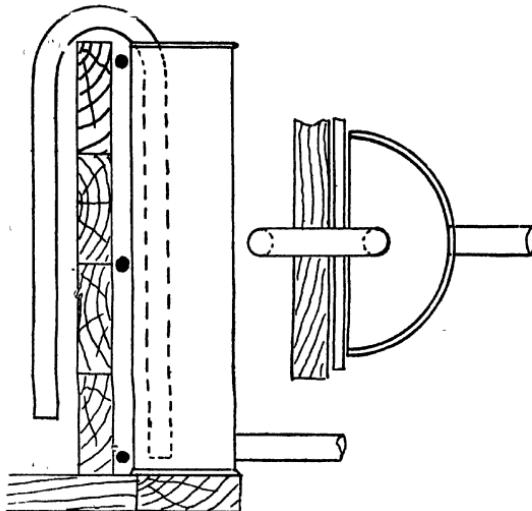


FIG. 51.  
Syphon Box for Acid or Mother Liquor Tank.  
FIG. 52.

handle is then covered with sheet lead and the joints carefully "burned" by the plumber. Either the plug and seat method or the syphon box method is suitable for the sulphuric acid, but the latter method is most suitable for the mother liquor, as it frequently happens that the pipe requires washing through with hot water to remove crystals. Around the plug is placed a large pipe made from sheet lead, the bottom portion of which is perforated with holes as shown in Fig. 50. This prevents bits of stick or dirt from getting into the pipe and taps. The provision of one of these methods is very necessary, as it enables any repairs to be done to the tap without emptying the tank. The pipe, *i* (Fig. 47), should be sufficiently

long to always remain sealed, otherwise offensive gases will enter the atmosphere.

In some works the mother liquor is fed into the saturator at stated intervals, and may then be elevated direct from the

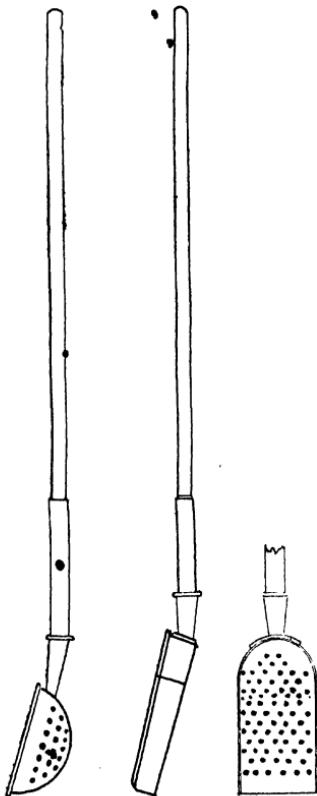


FIG. 53.  
Scoops for Hand Fishing.

FIG. 54.

underground drainage tank to the saturator by means of a regulus injector, but the writer much prefers, whenever possible, to supply it steadily and continuously as above described.

The gases which are not absorbed by the sulphuric acid are of a dangerous and evil-smelling nature, and are led away

## 64 MANUFACTURE OF SULPHATE OF AMMONIA

from the saturator along the pipe marked *j*, to be dealt with as described in Chapter X. In this pipe also a  $\frac{1}{2}$ -inch hole is drilled and closed with a tapered wooden plug in order that the gases may be frequently tested with red litmus paper to see that no ammonia is escaping.

As the sulphate of ammonia forms it flows down to the fishing pocket, from whence it is removed by means of copper scoops similar to that shown in Fig. 53, or by copper shovels

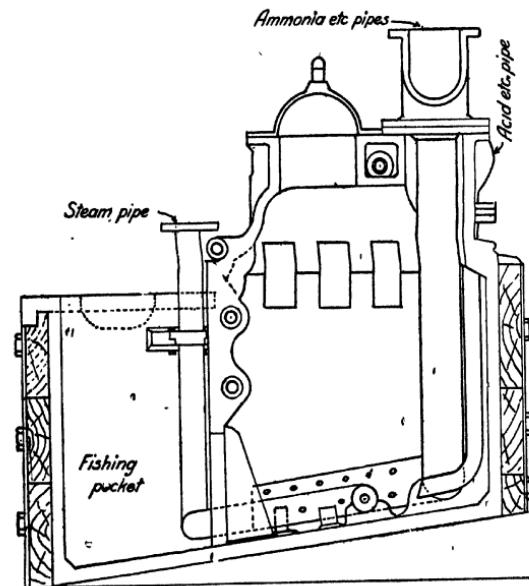


FIG. 55.—Plate-lead Saturator for Hand Fishing. Side Elevation.

like that in Fig. 54. In either case they are furnished with long pitch-pine handles, and if the latter form is adopted there may be a small cross piece at the top of the handle similar to that on an ordinary shovel.

Some of the neatest, most durable and efficient saturators are made from plate lead, varying in thickness from  $1\frac{1}{16}$  inch at the top to 2 inches at the bottom, stiffened by suitable brackets and bars and by external wood framing. Such a saturator, adapted for hand fishing, and made by Messrs Joseph Taylor & Co., is shown in Figs. 55, 56 and 57. To those who have carefully followed the details of the simple saturator

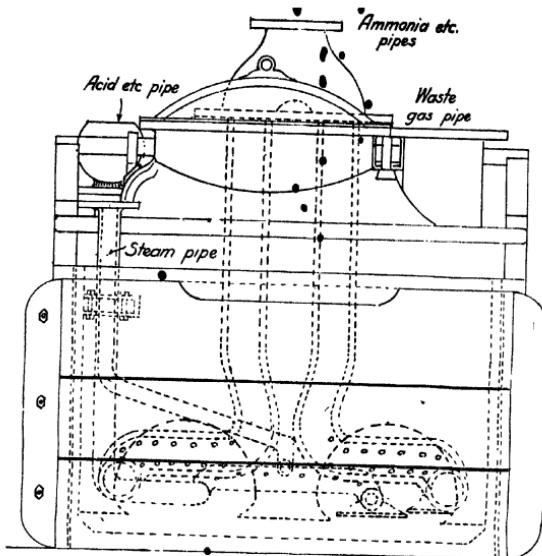


FIG. 56.—Plate-lead Saturator for Hand Fishing. Front Elevation.

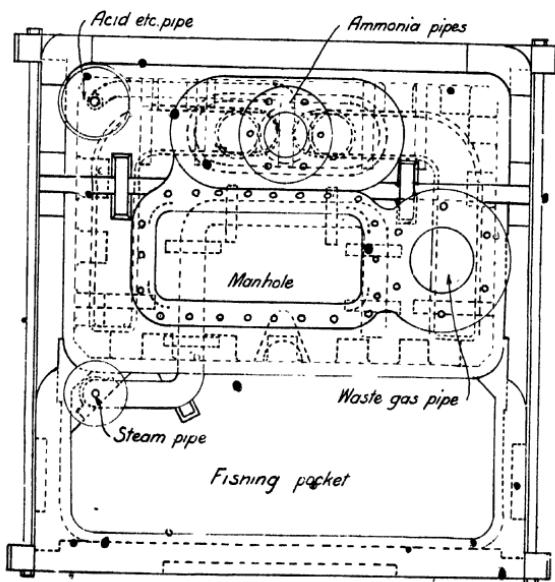


FIG. 57.—Plate-lead Saturator for Hand Fishing. Plan.

## 66 MANUFACTURE OF SULPHATE OF AMMONIA

already described, these more complicated drawings will not present much difficulty.

The ammonia pipe divides into two branches and in the bottom takes somewhat of a horse-shoe shape. By this means it secures very efficient distribution of the ammonia gas in a large number of small jets throughout the whole of the bulk of the mother liquor. It is also made readily detachable so that it can be removed without damaging the body of the saturator.

The acid, mother liquor and waste gas arrangements are similar to those in the simpler form.

One of the difficulties that the user of the saturator occasion-

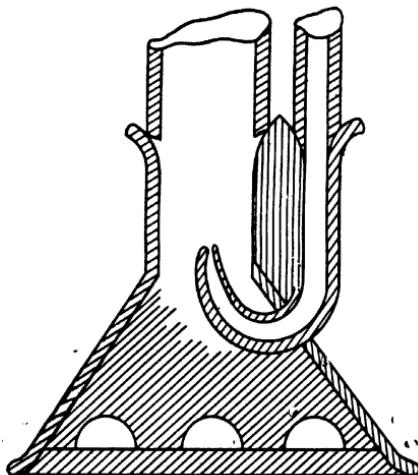


FIG. 58.—Cortin Improved Automatic Ejectors for Saturators.

ally meets is the tendency for the sulphate of ammonia to form a hard mass of small crystals over the ammonia pipe, especially during the time the plant is standing. On a nucleus thus formed rapid growth takes place until the pile is apt to reach above the level of the mother liquor, especially if the contents of the saturator have been worked down rather low. Such an occurrence is locally known as "hogging," and to prevent or remove it many manufacturers insert either hot-water or steam pipes, to use once or twice a shift, and especially on shutting down the plant. A most efficient form of this arrangement is shown in the figures now under consideration, and other uses of this useful pipe will be discussed in Chapter XI., when dealing with the working of the plant.

With reference to the durability of this type of saturator, Mr Taylor informed the writer in 1908 that one had been at work in Bolton for seven years without any repairs, that one had been at work at Croydon for ten years and would then last another three or four years, while one was still at work in

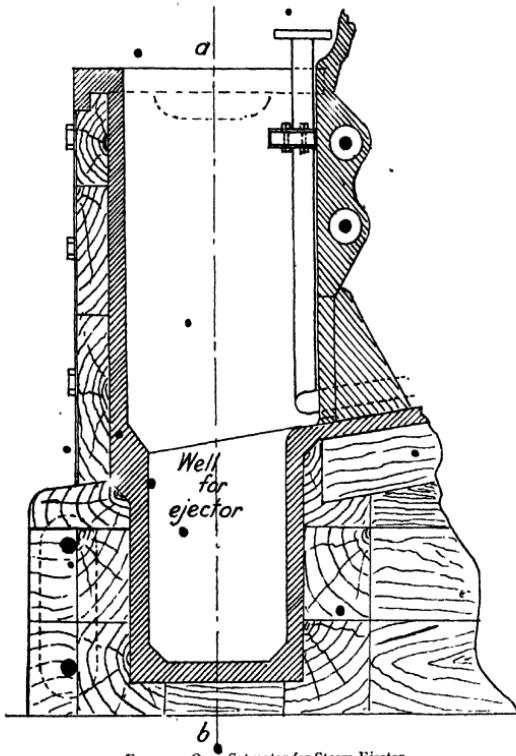


FIG. 59.—Open Saturator for Steam Ejector.

Lancaster that was put in fourteen years previously, and one at Southport only a year less.

The next type of saturator to be considered is that in which the sulphate of ammonia is removed from the saturator by means of a steam ejector, such as that made of regulus, shown in Fig. 58. There is also one shown *in situ* in Fig. 61.

The open type of this saturator is exactly the same as those

already described, except that the flat-bottomed pocket for hand fishing is replaced by a well-shaped pocket such as is shown in Figs. 59 and 60 in order to give the sulphate of ammonia a lead to the ejector.

We will now proceed to make acquaintance with the closed type of saturator. This is the most modern type, and though it possibly presents a little more difficulty to the mastering of its working by a beginner, its easier working afterwards, its

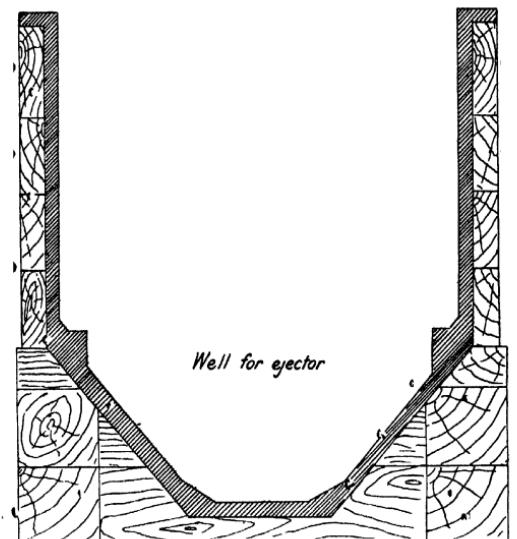


FIG. 60.—Open Saturator for Steam Ejector. Section on line *a-b*, Fig. 59.

greater freedom from smell and its great saving in labour compared with hand fishing, commends it to all practical men.

A circular saturator of this type, fitted with steam ejector and made of plate lead, by Messrs Joseph Taylor & Co., is shown in Figs. 61 and 62. In closed saturators, as there is not the open pocket in which to observe the depth of mother liquor present, and from which samples for testing may be obtained, these requirements are met by the insertion of a "seal" pipe as shown.

The steam ejector may discharge either on to the draining table or into little corves similar to those used in collieries, standing on or over a draining floor. More will be said on this subject in our next chapter.

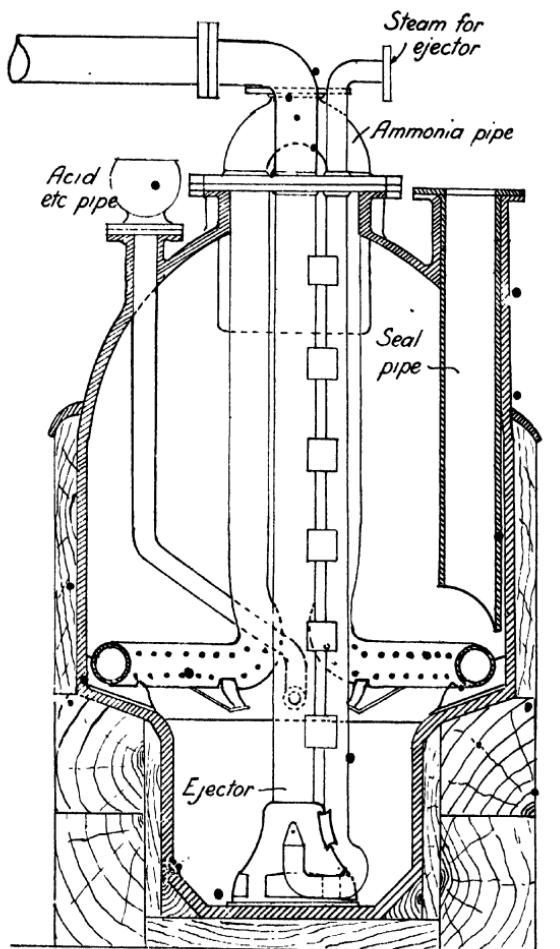


FIG. 61.—Plate-lead Circular Cylindrical Saturator, with Steam Ejector.

The next type of saturator to be considered is that having a bottom discharge valve, and this type certainly most nearly approaches the ideal arrangement.

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One of the earliest, if not the earliest, was brought out many years ago by the late Mr Alfred Colson, of Leicester, and though it has been in use for so long it still requires a lot of beating. It is shown in Fig. 63. The body consists of cast-iron sections lined with lead, the whole being securely bolted together. The valve (not shown in our drawing) is one of Mr Colson's many inventions.

A somewhat similar saturator made by Messrs C. & W. Walker, Limited, is shown in Fig. 64 (page 72), and a very

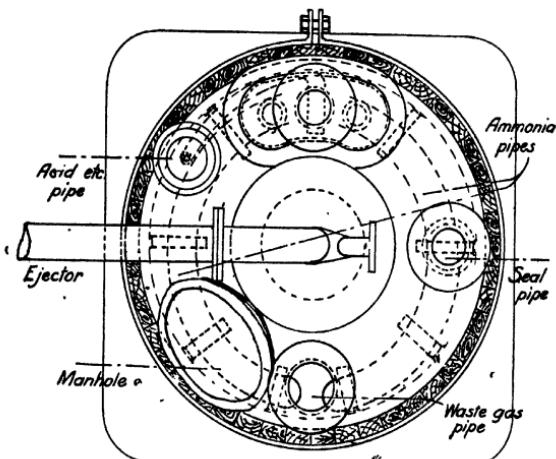


FIG. 62.—Plate-lead Closed Circular Saturator, with Steam Ejector.

effective valve for the bottom discharge made by them is shown in Fig. 65 (page 72).

Messrs Joseph Taylor & Co. also make this type of saturator in plate lead, and two of them in course of construction are shown in Fig. 66 (page 73).

Some trouble is experienced at times (especially through carelessness in starting up) through the ammonia pipes splitting along the line of the holes, and when this occurs it leads to local alkalinity, discoloration of the salt and loss of ammonia. To obviate this, Messrs Williams & Fenner have introduced a patent external ammonia pipe (or "cracker pipe"), shown in Fig. 67 (page 74).

Through various causes, such as an insufficient supply of acid, splitting of the ammonia pipe, "hogging," etc., it occasionally happens that a little ammonia passes the saturator, thus leading

## PLANT FOR MANUFACTURE OF SULPHATE 71

to loss. For a long time manufacturers have felt that if the waste gases from the first washing could be passed a second time

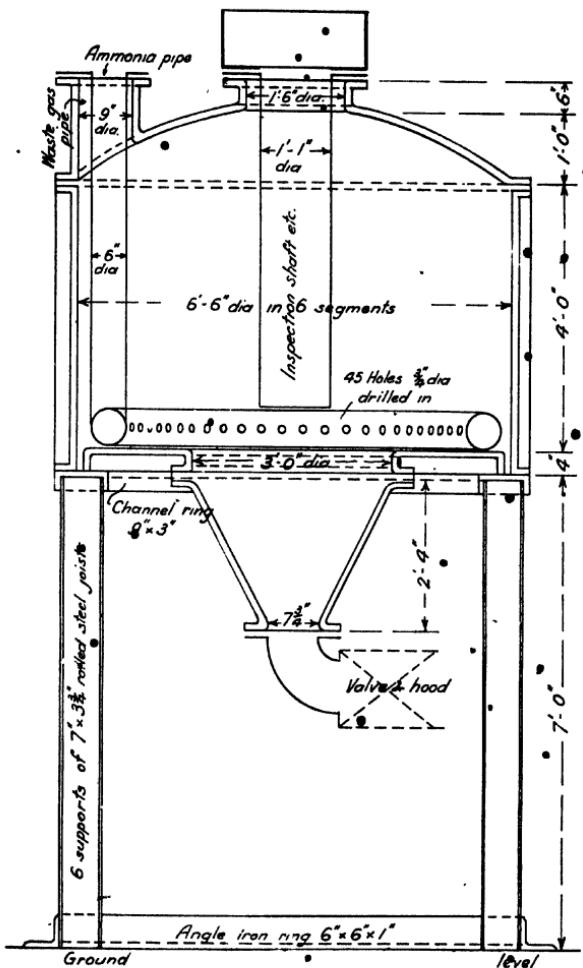


FIG. 63.—Colson Saturator. Section.

through the fresh acid on its way to the saturator it would be a very desirable end to accomplish, and Herr Karl Zimpell has

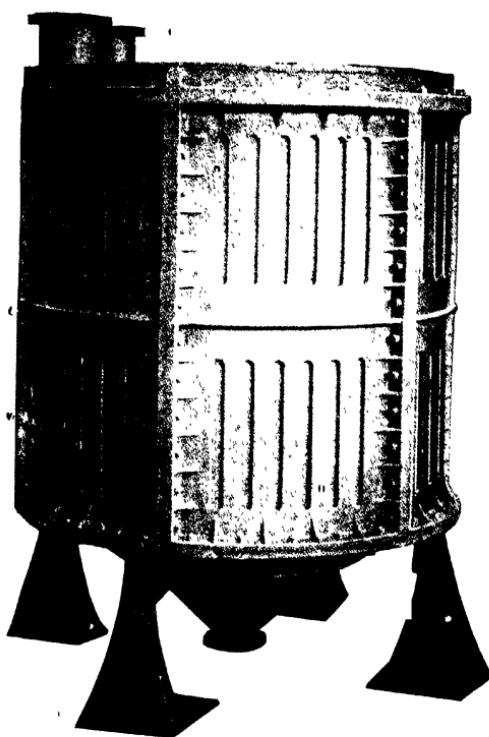


FIG. 64. —Walker Self-discharging Saturator.

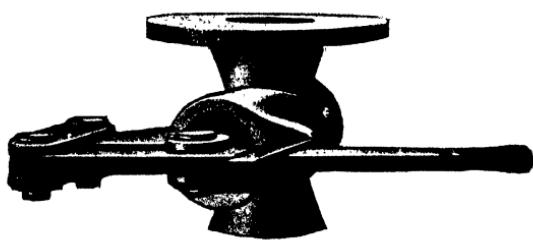


FIG. 65.—Improved Discharge Valve."

made a very laudable attempt to solve the problem in this direction. His patent saturator (British patent, No. 23,379, 1904) is shown in Fig. 68. The sulphuric acid enters through the pipe, *a*, fills the tray, *b*, and overflows through the pipe, *c*, into the main body of the saturator, *d*. The ammonia

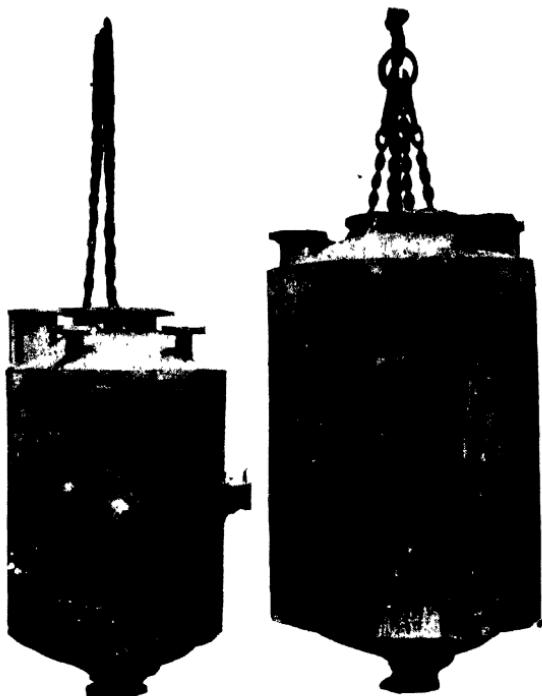


FIG. 66.—Taylor & Co. Saturator.

enters by the pipe, *e*, and is absorbed in the usual manner in the main body of the saturator, *d*. Any that should escape passes with the waste gases under the serrated hood, *f*, and meets the incoming acid a second time in the tray, *b*, where it is absorbed. The waste gases pass away up the pipe, *g*.

While the main lines of this patent are very commendable, there appear to the writer to be two grave defects in its construction. In the first place, the open-ended ammonia pipe is

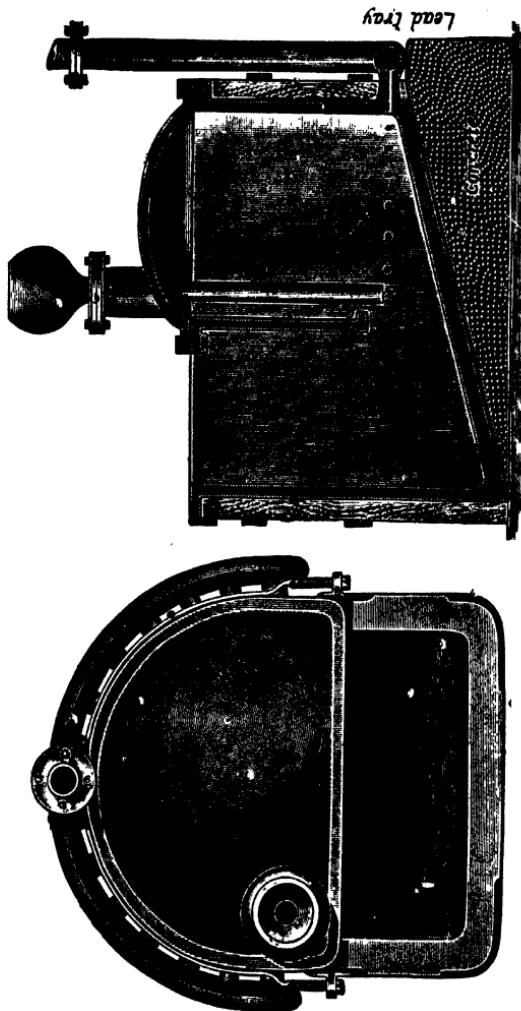


Fig. 67.—Williams & Fenner Patent Outside Cracker Pipe. Section.  
Plan.

almost certain to lead to local alkalinity, and this would be likely to throw such a strain upon the absorption of the upper chamber as would more than neutralise any advantage gained

from it. This defect could readily be remedied by continuing it as a circular pipe round the inside of the bell near its base, securing it by strong straps to the bell, and piercing it with a suitable number of holes, blowing towards the centre.

The second difficulty will be the matter of effecting any repairs in the upper chamber of the bell, and it would be difficult to remedy this and at the same time retain it as an integral

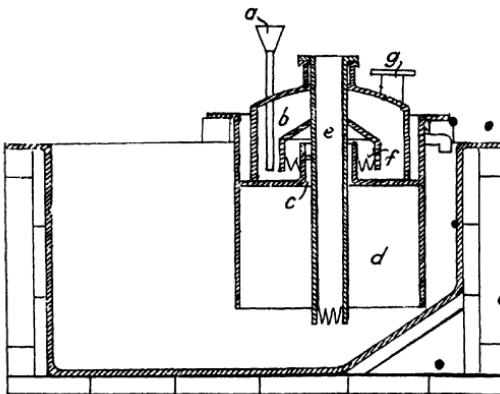


FIG. 68.—Zimpell Patent Saturator.

part of the saturator. Probably the best place for it would be as an accessory of the saturator placed in the waste gas pipe leading from it, and this arrangement is embodied in Feldman's saturator (British patent, No. 11,525, 1902).

Messrs S. Barnett & Co., Limited, have introduced a method by which steel plates may be coated with lead to any thickness from about  $\frac{3}{2}$  inch upwards, the union of the two metals being practically homogeneous. Such a material opens up many possibilities in saturator construction, and is well worth consideration by the makers.

## CHAPTER IX

### PLANT REQUIRED FOR THE MANUFACTURE OF SULPHATE OF AMMONIA (*continued*): DRYING, STORING, PACKING, ETC.

As the sulphate of ammonia forms in the saturator it settles down to the lowest point. In the case of the hand-fished saturator this is the fishing pocket, and from thence it is removed by means of the scoop or perforated shovel shown in Figs. 53

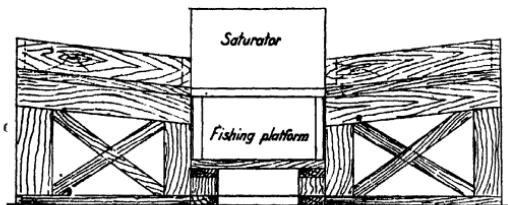


FIG. 69.—Draining Table. Front Elevation.

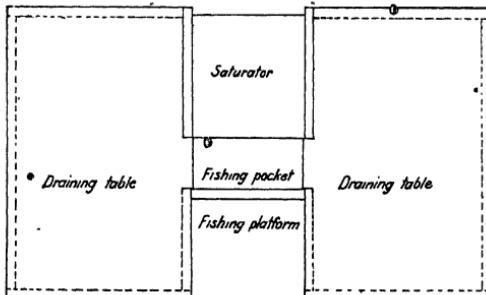


FIG. 70.—Draining Table. Plan.

and 54 in our previous chapter. It is then placed on a draining table such as is shown on each side of the saturator in Figs. 69 and 70. This table is built of 9-inch  $\times$  3-inch red-wood and placed on suitable supports as shown. The border is formed of 9-inch  $\times$  3-inch planks on edge, except across the end of the

fishing pocket. The whole is lined with lead of about 10 lbs. per foot strength, and the portion across the end of the fishing pocket is turned over that portion of the saturator to form a gutter to allow the mother liquor to drain back into the saturator.



FIG. 71.

To protect the lead, and also to facilitate the drainage of the salt, pieces of pitch pine, 3 inches  $\times$  3 inches, the lower edges cut out as shown in Fig. 71, are laid upon the lead work about a foot apart, and on these are placed pitch-pine boards, 6 inches  $\times$  1 inch, and on these boards the salt is placed as it is fished out by the scoop. From there at suitable intervals it is thrown on to the floor of the sulphate store, and after standing there for some time for further drainage it is stacked on the heap in

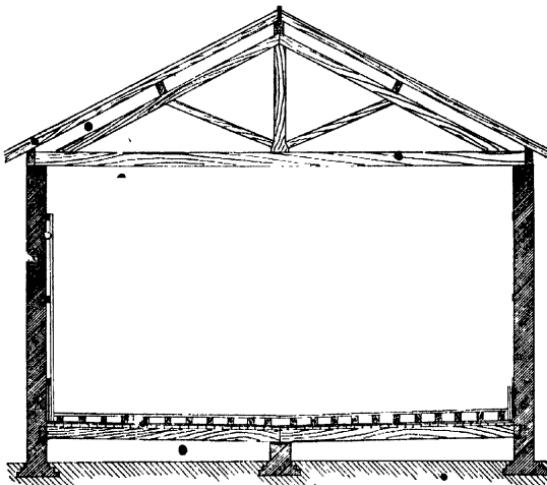


FIG. 72.—Sulphate of Ammonia Storeroom.

the store. It is advisable to only stack the sulphate of ammonia across half the width of the store at a time, as by this means one-half is getting in condition for packing, or may be packed, while the other half is being filled.

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A section of a sulphate of ammonia store is shown in Fig. 72 (page 77). The floor is formed of suitable joists, a slope of about 4 inches being given from the sides to the centre, and a similar slope from back to front, to carry off the mother liquor draining off from the salt. On these joists are nailed 1½-inch tongued and grooved flooring boards, care being taken to punch the nails well home, as even a slight projection of a nail head is sure to cause trouble later, by cutting a small hole through the overlying lead. On these boards is laid a covering of about 8 lbs. lead, the edges being turned up about 2 feet against the walls

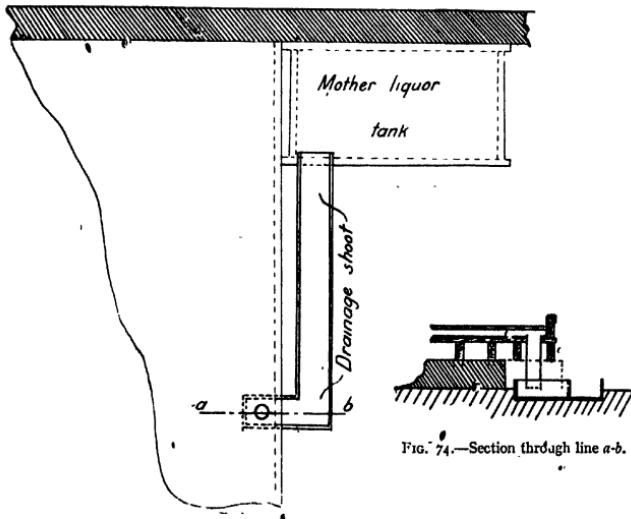


FIG. 73.—Plan of Sulphate of Ammonia Storeroom.

of the room to form a dish, the joints in all cases being carefully "butfned." The walls are then lined with 1½-inch tongued and grooved flooring boards about 10 feet high as shown, the upstand of the lead dish being between these boards and the wall. The drainage from this floor passes through a 6-inch pipe to an open wooden trough lined with lead, which conveys it to the mother liquor tank as shown in Figs. 73 and 74. On the lead floor are placed pieces of pitch pine, 3 inches × 3 inches, as seen in Fig. 71, and on these are laid loosely red-wood boards, 6 inches × 1 inch. These need not be planed nor tongued and grooved, as the mother liquor requires to drain through the joints, and they have to be taken up frequently for

## PLANT FOR MANUFACTURE OF SULPHATE

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cleaning. The time that the salt will require to lie in store before being in fit condition for packing will necessarily vary somewhat,

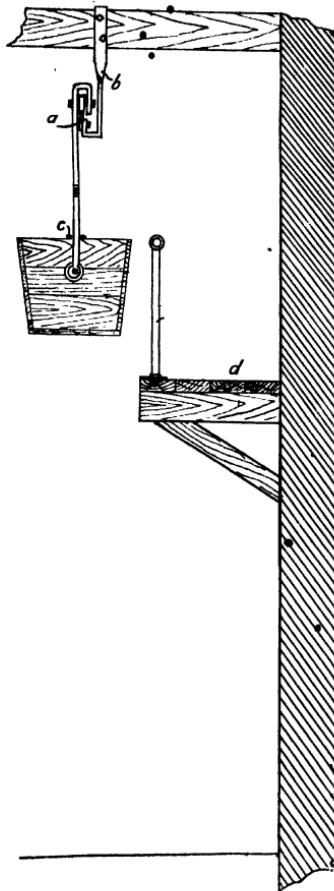


FIG. 75. -Overhead Corves.

but if the draining floor is in clean condition (*i.e.* free from crystals between the draining floor and the lead), and the salt is in normal condition on leaving the saturator, about three to four days should suffice.

## 80 MANUFACTURE OF SULPHATE OF AMMONIA

In all cases the tanks for the mother liquor should be of ample size, as nothing is more annoying than to find on starting the plant, or anything going wrong, that we are crippled by lack of storage room for this product. The total storage for mother liquor should be *at least* three times the capacity of the saturator.

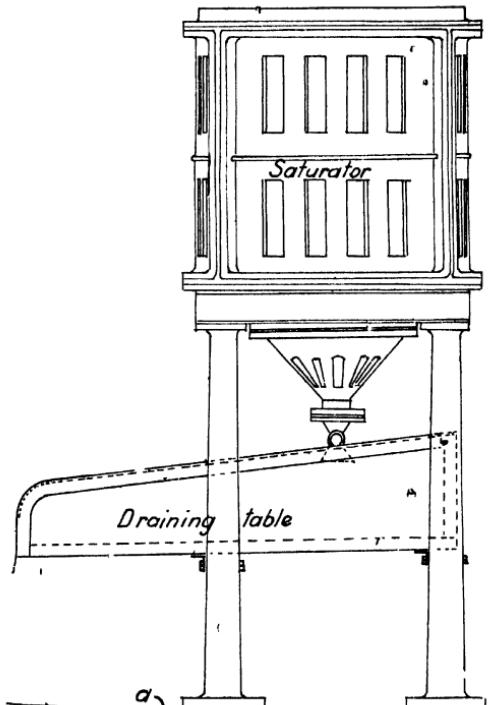


FIG. 76.—Draining Table for Saturator with Bottom Discharge.

When the salt is removed from the saturator by means of a steam ejector, the draining table is similar in construction to those shown in Figs. 69 and 70, but in this case only one table is provided. If the salt is discharged on to the floor of the draining table, the sides near the discharge pipe of the ejector are made much higher to receive the splash.

Instead of the discharge taking place on to the floor of the draining table, it may be made into curves similar to those used

in collieries, except that, as the wheels do not run upon rails, they are made with plain tyres. These are pushed under the discharge pipe as required, and after being filled, are allowed to stand on the draining table for some time to drain, after which their contents are emptied either into the hydro-extractor (where these are used) or into the store. In cases where the plant is worked both night and day, and a hydro-extractor is employed, it is scarcely necessary to point out that if sufficient corves are provided to hold the night production it will save

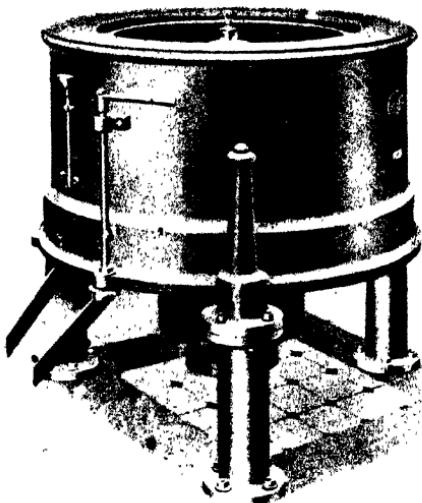


FIG. 77.—Suspended Electrically-driven Hydro-Extractor with Bottom Discharge.

keeping a man to work the "hydro" during the night, as the day man can then deal with the whole production.

Where the levels suit, these corves may run along an overhead rail, as shown in Fig. 75. In this arrangement the running rail, *a*, is formed of 4-inch  $\times$   $\frac{1}{2}$ -inch flat-iron on edge, carried on hangars, *b*, attached to the roof timbers. The corve is swung

in a  $\perp$  shaped handle and the corve is prevented from tipping over until required by the hinged catch, *c*. The men go along the gangway, *d*, to push the corves, and the handrail on stanchions, *e*, protects them.

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The draining table suitable for saturators of the bottom discharging type is shown in Fig. 76. The table is lined with lead, and covered with the loose floor as in previous cases. The mother liquor in this case runs on to the floor, *a*, which is lead-covered and dished, and is also covered with the usual loose floor, from which it is conveyed by suitable means to the mother liquor storage tank. In cases where this draining table comes over the hydro-extractor for convenience in charging the latter, to prevent the drainage passing into the "hydro" the lead

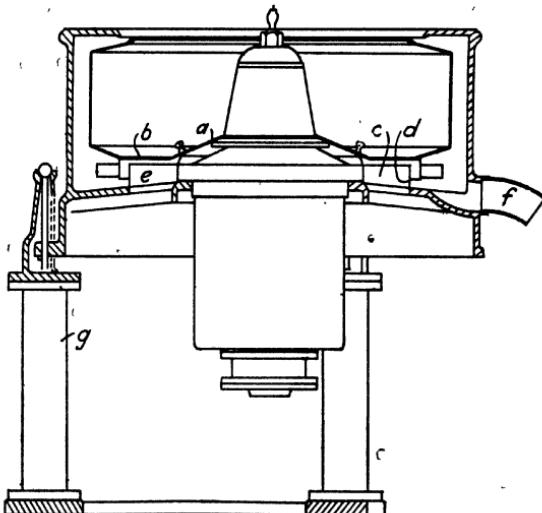


FIG. 78.—Suspended Electrically-driven Hydro-Extractor with Bottom Discharge.

near the "hydro" end of the draining table is turned up about 2 inches and the corners "burned," the drainage being led away to the floor below by means of a lead pipe from 2 inches to 3 inches in diameter.

The mother liquor which drains from the various draining tables and floors (except from those above the saturator level when it drains back into that vessel) is collected into the basement tank or tanks, and is elevated into the tank supplying the saturator, either by means of an acid elevator such as that already shown in Figs. 30 and 31 or by means of a regulus metal steam injector.

In cases where, either from limited storage room or other

## PLANT FOR MANUFACTURE OF SULPHATE 83

causes, it is desirable to get the sulphate of ammonia packed into bags as soon as possible after being made, the most expeditious method of drying the salt is that by means of hydro-extractors (also known as "centrifugals"), various forms of which are shown in Figs. 77 to 81. They consist essentially

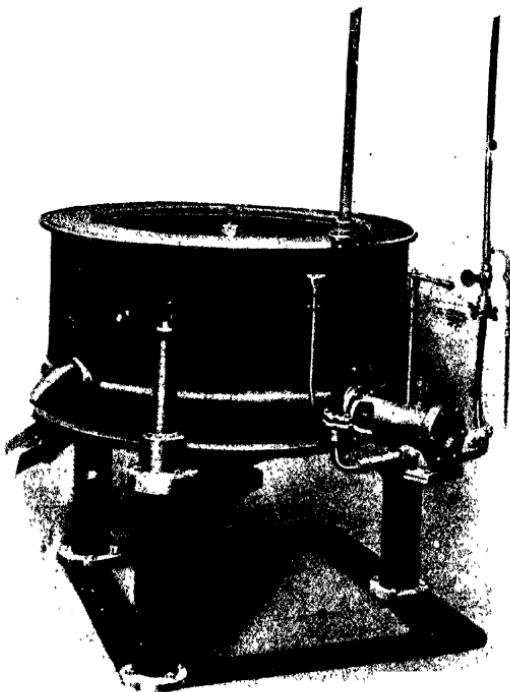


FIG. 79.—Suspended Direct Steam-driven Centrifugal.

of an outer cast-iron circular vessel inside which is a copper basket, the sides of which are perforated with a large number of small holes. This basket is filled to a convenient depth with the sulphate of ammonia raked in from the draining table or tipped in from the corves. The motor or steam engine is then started and the basket revolves at a very high speed, thus, by centrifugal force, forcing the salt against the sides of the

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basket and expelling the moisture through the holes in the basket into the outer casing, through the outlet of which it finds its way on to the drainage floor beneath. The time required to effect this drying will vary according to the condition of the salt as put into the "hydro," but will usually range from  $2\frac{1}{2}$  to 4 minutes. The most suitable time must be fixed by experiment, basing it upon analysis of the moisture in the dried sulphate of ammonia ascertained as directed in Chapter I. Fig. 77 shows an electrically-driven hydro-extractor

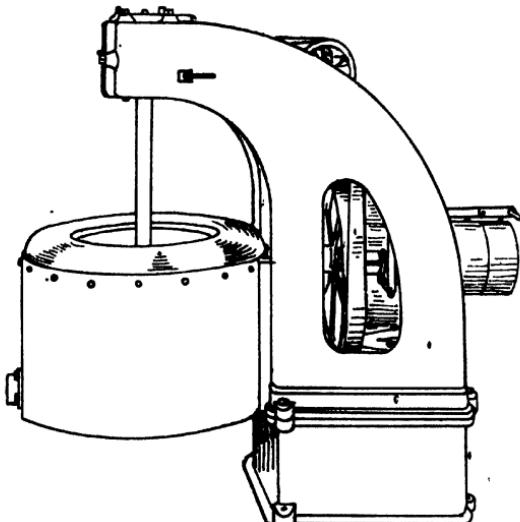


FIG. 80.—Patent Self-balancing Centrifugal ("Weston" Type) Swan-neck Pattern.

made by Messrs Thomas Broadbent & Sons, Limited, and Fig. 78 shows a section through the same. The copper basket is shown at *a*, while *b* is the discharging door secured by the bolt, *d*. The salt is discharged through the shoot, *c*, *e*, while the mother liquor escapes through the pipe, *f*. The height of the standard, *g*, is altered according to the requirements of the situation. The discharging arrangements can also be readily modified to suit the surrounding plant. Fig. 79 shows the steam-driven type made by the same firm, the internal arrangements being similar to the one already described. Fig. 80 shows a form made by Messrs Watson, Laidlaw & Co., Limited, and Fig. 81 one of the types manufactured on the Continent.

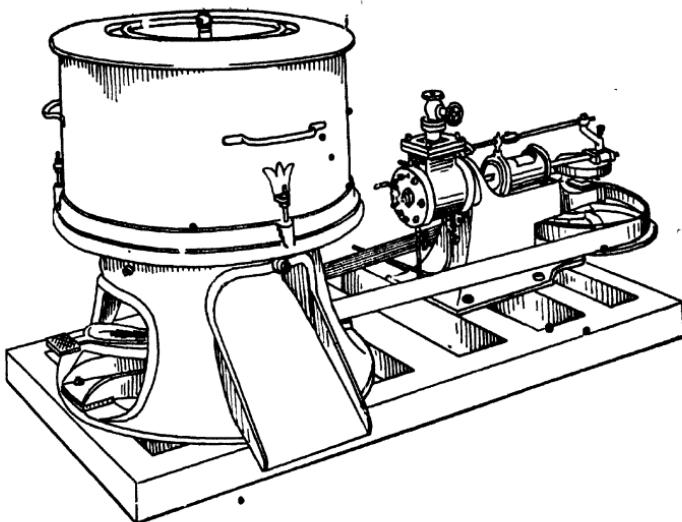


FIG. 81.—Centrifugal with Bottom Discharge.

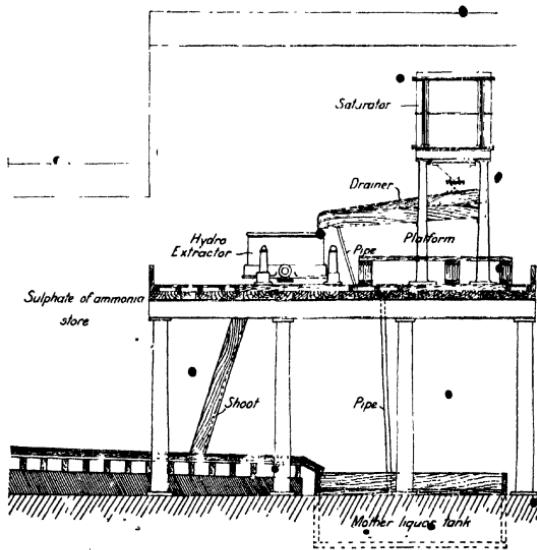


FIG. 82.—Saturator, Draining Table, Hydro, etc.

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An ideal arrangement of saturator, draining table, "hydro," etc., is shown in Fig. 82 (page 85), and from practical experience with such an arrangement the writer can highly recommend it.



FIG. 83.—Simon Patent Automatic Sack-filling and Weighing Machine.

The shoot from the "hydro" may lead directly to the hopper of an automatic weighing machine or the mouth of a bag holder, but this is an arrangement that does not commend itself to the writer, as he finds, from experience, that if the salt is bagged immediately after leaving the "hydro" it sets

very hard in the bags, and leads to trouble and tearing of the bags in subsequent handling for shipment.

If the salt is allowed to stand a short time at the base of the shoot from the "hydro," and is then turned over to a heap it is a great advantage, and packing from this heap may begin a few hours after the salt is placed there.

In packing the sulphate of ammonia, machines are supplied which hold the bag and automatically cut off the supply when the contents of the bag reach a certain weight. Such a machine

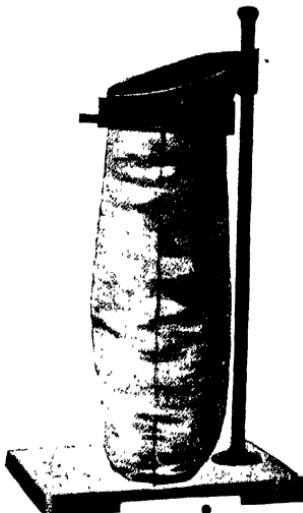


FIG. 84.—The "Phoenix" Patent Sack Holder.

is shown in Fig. 83. This machine is made by Messrs Richard Simon & Sons, Limited. A machine to answer the same purpose has also been designed by Messrs W. & T. Avery, Limited.

In cases where the sulphate of ammonia manufacturer desires to weigh on the ordinary sack-weighing machine, for the filling the writer strongly recommends him to obtain one of the patent mechanical sack holders, and he can confidently say that anyone who has once used them would never dream of reverting to the old-fashioned method of holding the sack by hand. Not only do they save the services of the man holding the sack, but also the loss of time caused by the "filler" having

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to "come to the assistance" of the "holder" from time to time to shake down the contents of the sack. Such a holder is made by Messrs Richard Simon & Sons, Limited, and is shown in

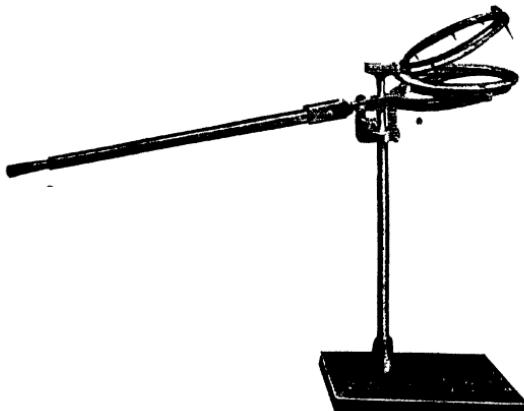


FIG. 85.—Dunn Patent Sack Holder.

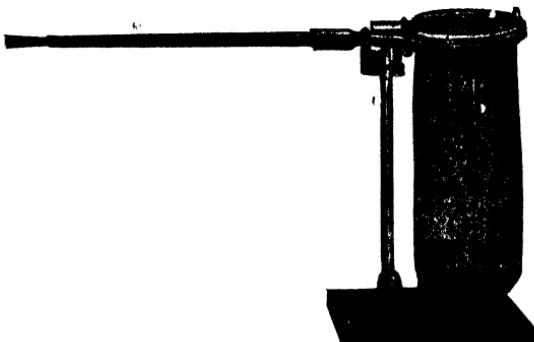


FIG. 86.—Dunn Patent Sack Holder.

Fig. 84. The bag is attached to the mouthpiece of the holder by means of a leather strap, which is rapidly tightened up by means of a short lever. Another design for accomplishing the same end, shown in Figs. 85 and 86, is made by Messrs Hattersley & Davidson, Limited.

The bags used for the packing of sulphate of ammonia are chiefly second-hand ones that have been used for the carriage of wheat or sugar, and for greater security, of late years buyers frequently request that the salt shall be packed in double bags. In this case the smaller inner bag should be placed inside the

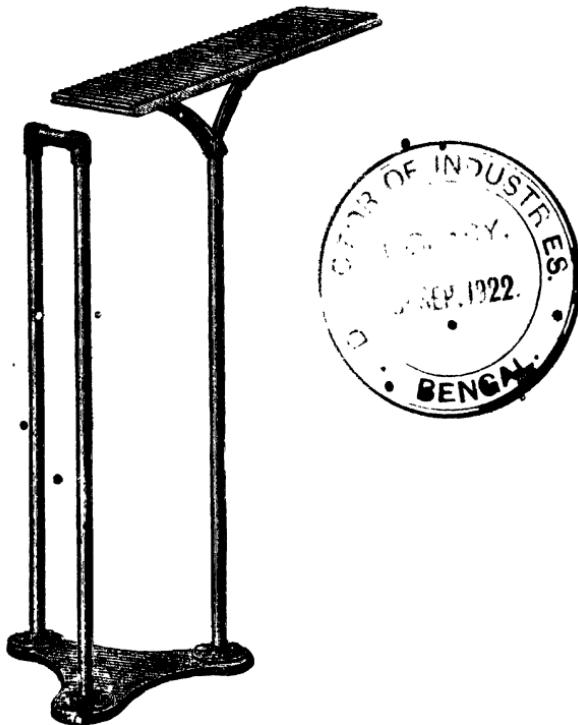


FIG. 87.—Hattersley & Davidson's Bag-Doubling Apparatus.

larger outer one *before* filling, as it is most difficult to do so afterwards. It is not easy to double the bags without the aid of some mechanical appliance, and for this purpose Messrs Hattersley & Davidson, Limited, supply the contrivance shown in Fig. 87. A piece of home-made apparatus for the same purpose, used by the writer, is shown in Figs. 88 and 89. It is made of an elm board slightly narrower at the top end than

## 90 MANUFACTURE OF SULPHATE OF AMMONIA

the smallest bag and tapering about 2 inches to the bottom end. It is slightly longer than the longest bag and is let into the base board with a tenon and mortice joint. Its edges are slightly

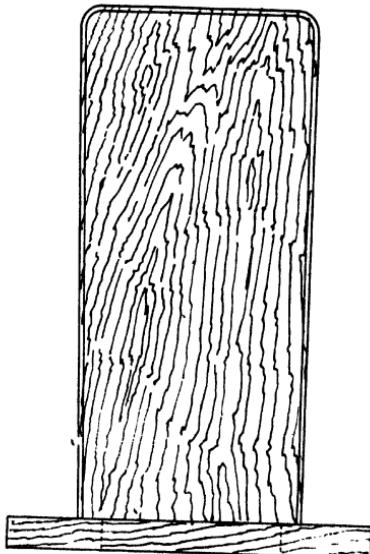


FIG. 88.



FIG. 89.

A Home-made Bag-Doubling Apparatus.

rounded. The base board is secured to the floor by coach screws, or if it is preferred to use the apparatus horizontally, it may be secured to the wall. The method of using is obvious.

If the string used for the sewing of the bags is purchased, ready cut into 6-foot lengths, it will be found to be a convenience.

## CHAPTER X

### PLANT REQUIRED FOR THE MANUFACTURE OF SULPHATE OF AMMONIA (*continued*): THE WASTE GASES—CONVERSION INTO SULPHURIC ACID—CLAUS PROCESS—OXIDE PURIFICATION, ETC.

THE gases from the still that are not absorbed by the sulphuric acid in the saturator are known as "the waste gases." They consist very largely of carbon dioxide, sulphuretted hydrogen and moisture, with a little pyridine, cyanogen compounds, and many other substances, the whole forming a very evil-smelling and highly-poisonous compound. They leave the outlet pipe of the saturator, and after passing through the baffle box, pass through the superheater, as described in Chapter III., where they heat up the ammoniacal liquor to a temperature of about 200° Fahr. When the waste gases leave the superheater, they are still much too hot and damp for the subsequent treatment, so it is necessary to cool them still further. A simple form of apparatus for accomplishing this purpose is shown in Fig. 90. A series of thin cast-iron pipes is led backwards and forwards, and over them is placed a 2-inch or 3-inch wrought-iron water pipe pierced on its under surface with small holes. This is connected with a suitable water supply, and by means of a cock the supply is adjusted to secure the cooling of the gases to the temperature of the air.

A very efficient but more elaborate system of condensers is shown in Fig. 91 (page 93). This was used by the writer some years ago in a works over which he had charge and gave every satisfaction. It consisted of a series of three condensers of the battery type arranged horizontally, each one of which contained 79 2-inch tubes. The waste gases were conveyed from the superheaters along a long length of 12-inch cast-iron pipes and entered the shell of the top condenser as shown in the illustration. After passing downwards through the second and third condensers, they passed through two sets of 3-foot diameter cast-iron pipes arranged as shown. At first these were filled with large pieces of Durham coke, but as this led to choking and consequent back pressure the coke was eventually removed and the 3-foot pipes were worked without any packing whatever. The cooling was assisted by cold water which entered

the bottom battery condenser through a suitable valve, and

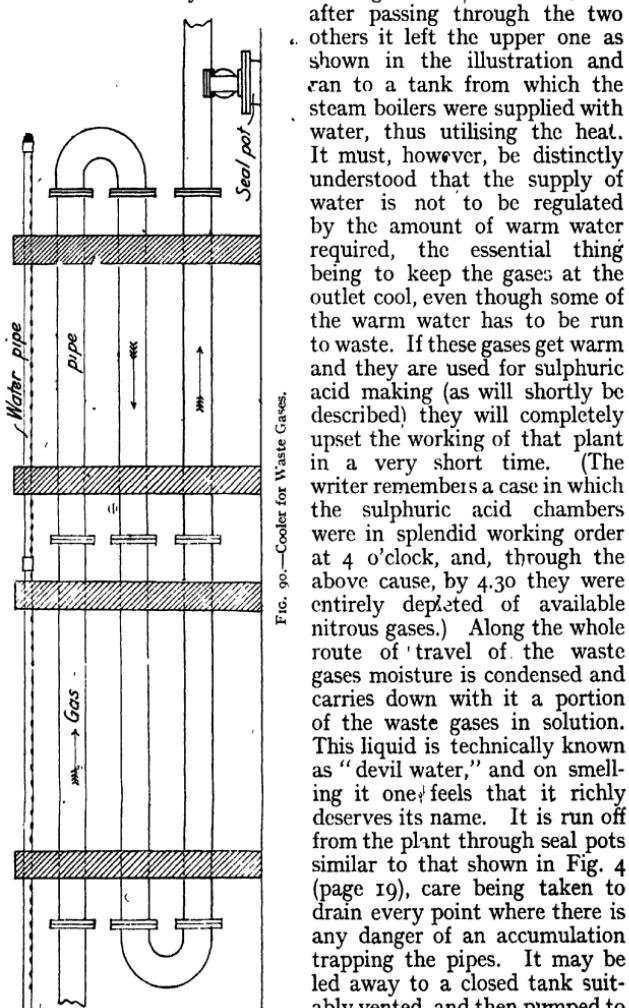


FIG. 90.—Cooler for Waste Gases.

after passing through the two others it left the upper one as shown in the illustration and ran to a tank from which the steam boilers were supplied with water, thus utilising the heat. It must, however, be distinctly understood that the supply of water is not to be regulated by the amount of warm water required, the essential thing being to keep the gases at the outlet cool, even though some of the warm water has to be run to waste. If these gases get warm and they are used for sulphuric acid making (as will shortly be described) they will completely upset the working of that plant in a very short time. (The writer remembers a case in which the sulphuric acid chambers were in splendid working order at 4 o'clock, and, through the above cause, by 4.30 they were entirely depleted of available nitrous gases.) Along the whole route of travel of the waste gases moisture is condensed and carries down with it a portion of the waste gases in solution. This liquid is technically known as "devil water," and on smelling it one feels that it richly deserves its name. It is run off from the plant through seal pots similar to that shown in Fig. 4 (page 19), care being taken to drain every point where there is any danger of an accumulation trapping the pipes. It may be led away to a closed tank suitably vented, and then pumped to mix with the ammoniacal liquor

supply (in which case the production can be accurately gauged, and any leakage of water into it from a plant like that shown

in Fig. 91 rapidly ascertained), or it may run straight to the ammoniacal liquor store, care being taken that the end of the pipe is under the surface of the liquor. In any case it is passed through the still again with the ordinary ammoniacal liquor, and this removes a considerable amount of the smell, thus enabling it to go away with the waste liquor without causing more than a minimum of nuisance.

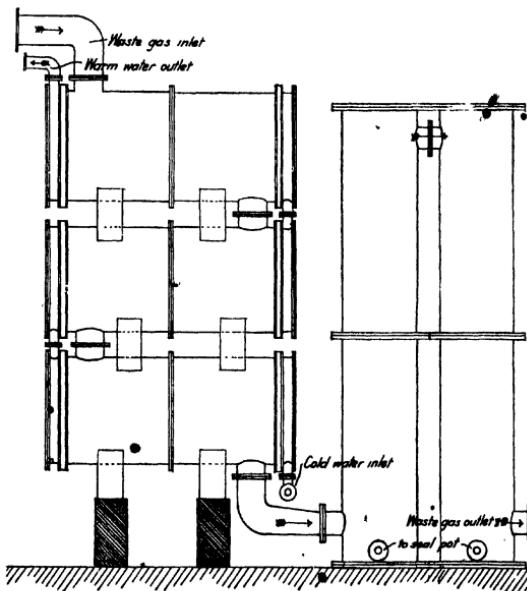


FIG. 91.—Coolers for Waste Gases.

After the waste gases are cooled and dried they may be dealt with in one or three ways, viz. :—

- (1) Converted into sulphuric acid.
- (2) Converted into sulphur by the Claus process.
- (3) Passed through an oxide of iron purifier.

The whole of the writer's personal experience has been in large works where sulphuric acid was also made, and where the first method was therefore practicable, and where this is the case it is undoubtedly the most satisfactory method of dealing with these gases, as it not only entirely removes any

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nusance caused by them, but by the sulphur in the sulphuretted hydrogen being used as a source of that material for the manu-

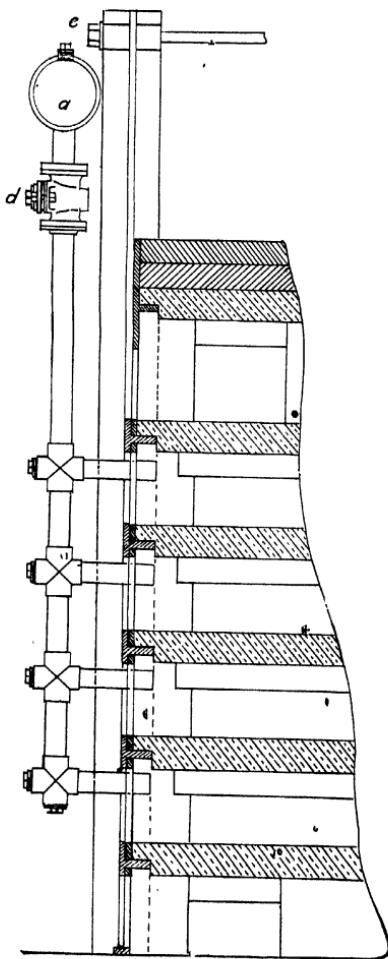


FIG. 92.—Handling Waste Gases.

facture of the sulphuric acid it materially decreases the "cost of producing this acid.

The waste gases are burned in the same kilns as the spent oxide or pyrite, the arrangements for conducting them into spent oxide kilns being shown in Figs. 92 and 93. The main pipe, *a*, is continued from the cooling plant, and at a point near the kilns the tee pipe, *b*, is inserted to act as a final catch for moisture. This is emptied from time to time, as

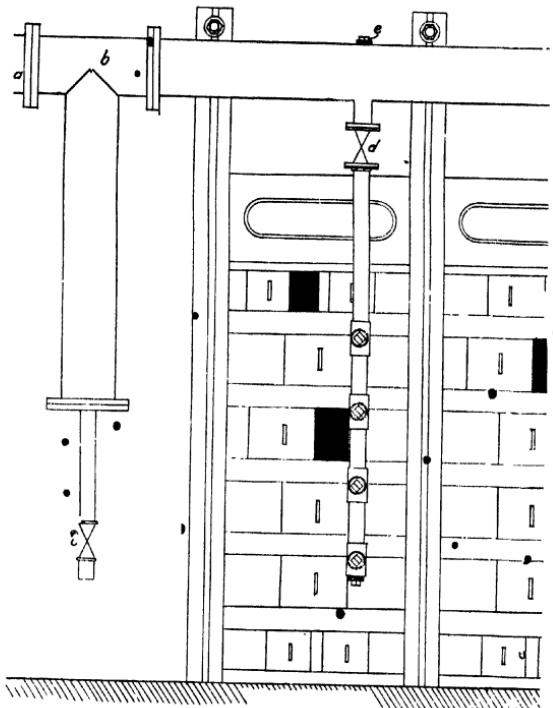


FIG. 93.—Hauling Waste Gases.

required, through the tap, *c*. The gases pass into each burner and each vertical section is controlled by a cast-iron cock, *d*. This cock is closed about half an hour before a spent oxide charge is charged in that section, and opened again about half an hour after the charging. The tubes below the tap will require cleaning out every two or three weeks, and this is most conveniently done when the sulphate of ammonia plant is standing. A good help to cleaning them out is afforded by soaking some

cotton waste in paraffin and igniting it in one or more of the crosses of a section and afterwards cleaning the pipes by means of an iron rod with a small disc at the end. Of course the tap of the section is closed while this is being done. Finally, the plug, *e*, is removed, the cock, *d*, momentarily opened and a rod passed right down as quickly as possible, then withdrawn, and the plug made good. The plugs are simply well screwed home and have no cementing material on them at all.

In cases where the quantity of waste gas is small and it is desirable to concentrate the heat, the gas is led into the bottom burner of each series only. The little alteration necessary to accomplish this is obvious.

There are only three slight objections to dealing with the gas in this manner, the first being that every 34 lbs. of sulphuretted hydrogen in burning produces 18 lbs. of water, which tends to reduce the strength of the sulphuric acid formed, but where the proportion of sulphuretted hydrogen compared with the spent oxide used is but small, this is not a serious matter.

The second objection is that the  $\text{CO}_2$  in the waste gases takes up a certain amount of the chamber space in the acid chambers; but even with a large proportion of sulphuretted hydrogen the writer has had no difficulty in working with as low a chamber space as 16 cubic feet per lb. of sulphur per twenty-four hours.

The third objection is that on ammonia escaping from the saturators, or the gases getting too warm, they upset the working of the chambers, but in this case the remedy is obvious.

These objections are very slight and scarcely worth mentioning in comparison with the advantages to be gained by this method of dealing with the gas. In addition to the advantages already mentioned, the gases are of great assistance in increasing the temperature of the kilns, thus securing better burning off of the sulphur in the spent oxide and even making it practicable to use a spent oxide of low percentage of sulphur.

The second method of dealing with the gases is that known as the Claus process. The writer has not had personal experience of this method, but those who use it speak highly of its efficiency.

The plant made by Messrs C. & W. Walker, Limited, is shown in Fig. 94, and their lucid description of the plant and its working is appended:—

“The plant consists of the following parts:—(1) A cast-iron atmospheric condenser for cooling the gases. (2) A dry coke scrubber, to eliminate any moisture remaining in the cool gases. (3) A kiln, lined with fire-bricks, and having a fire-brick grate on which rest loose broken fire-bricks, and over this is a layer of hydrated oxide of iron. (4) A cast-iron air inlet box attached

PLANT FOR MANUFACTURE OF SULPHATE

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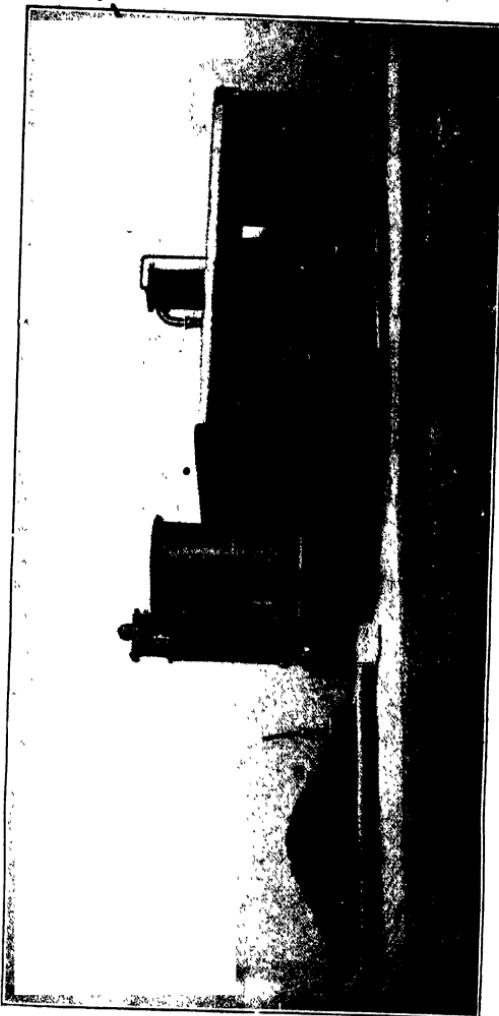


Fig. 94.—Claus Sulphur Recovery Plant.

to the upper portion of the kiln, in which the air and foul gases mix before entering the kiln. (5) An air engine, with self-regulating blow-off valve, to give a regular and continuous

G

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supply of air to the kiln. (6) A depositing or settling chamber, made of ordinary brickwork, the portion nearest the kiln being lined with fire-brick. This chamber contains numerous transverse baffle walls, to retard the flow of gas and the finely-divided sulphur, and encourage the deposit of the latter. The chamber is covered with slate slabs, and the outer walls have numerous openings for cleaning out the deposited sulphur. (7) A scrubber filled with limestone, down which a small stream of water is constantly flowing. This scrubber arrests any sulphurous acid gas which may be passing away with the waste-gases from the depositing chamber. (8) An open purifier made of brickwork or cast-iron, filled with hydrated oxide of iron to arrest any sulphuretted hydrogen which may be passing away with the waste gases from the limestone scrubber.

"**NOTE.**—The two latter portions of the plant (scrubber and purifier) are not necessary for the perfect working of the process, but act as catch vessels in the case of careless working, or when by-passing the plant.

"The acidity at the outlet of the depositing chamber must be less than three 3 grains per cubic foot.

"The action of the plant may be briefly described as follows:—The hot waste gases from the saturator pass through the heater of the sulphate of ammonia plant, and are partially cooled by the liquor before entering the still; they then pass through the atmospheric condenser, where they are cooled to the surrounding temperature. In cooling down, water is condensed which contains a large percentage of  $H_2S$ , and is very liable to cause nuisance in the neighbourhood if discharged into the drains. This water may be pumped back into the top of the still, and in passing through it gives off its  $H_2S$ , which goes forward through the saturator and into the kiln. The water is then perfectly inodorous, and passes away with safety into the drains with the spent liquor.

"The gases, after being thoroughly cooled in the condenser, pass into the air inlet box of the kiln, and mix with the air pumped in by the air engine. The mixed gas and air then enters the kiln at the top, passing downwards through a layer of hydrated oxide of iron, when the ordinary reaction takes place. The oxygen of the air supply uniting with the hydrogen of the  $H_2S$  forms water, leaving the sulphur to unite with the iron and form sulphide of iron, which is immediately re-oxidised by the air. This reaction takes place so rapidly that the whole mass of oxide is soon in an incandescent state; at this heat the sulphur produced by the reaction is volatilised and passes with the other gases through the fire-brick grating at the bottom of the kiln, into the depositing chamber, where it cools and settles down. The water spray formed by the reaction is thrown down

into the depositing chamber, where it drains away through the bottom brickwork of the side walls. The outlet gases from the depositing chamber pass through the wet limestone scrubber and the oxide purifier, and then escape into the air perfectly freed from all noxious gas.

"Should there be an excess or deficiency of air delivered into the kiln with the foul gases, the outlet gas of the chamber will contain traces of sulphurous acid, or sulphuretted hydrogen, which are taken up, respectively, by the limestone scrubber or the oxide purifier.

"The amount of marketable sulphur recovered should reach 90% of the sulphur passing into the kiln, and be of a very pure quality.

"The process may be carried on without any attention beyond what is required for the sulphate of ammonia making."

In order to test the exit from the Claus process, an aspirator (Fig. 95) is provided, made of sheet lead, 12 inches square and either 1, 2, or 3 feet in depth. The top is provided with a plug and socket, through the latter of which it may be filled with water, also a short brass tube to connect with the absorption apparatus. The bottom has a brass cock for running off. The front is provided with a glass tube to act as a water gauge, so that, if less than the whole contents of the box be run the necessary calculation may be made. Two 8-oz. bottles are provided with india-rubber stoppers and glass tubes inserted as shown. One end is connected with the aspirator, the other with the exit from the Claus kiln. The bottles are charged with 10 c.c. of hydrogen peroxide (10 volumes quality) and water added. The aspirator has previously been filled with water, and on turning on the bottom tap the quantity of water running

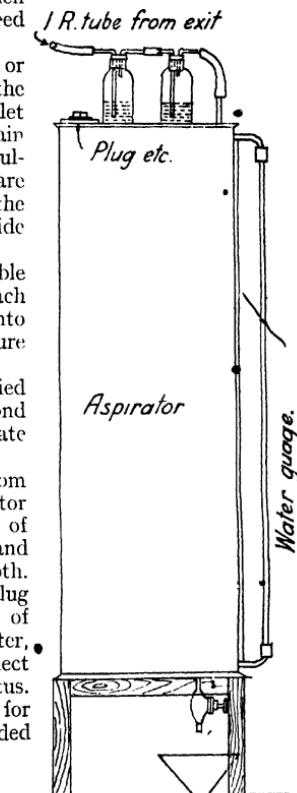


FIG. 95.—Apparatus for Testing Exit.

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out aspirates the same quantity of gas through the hydrogen peroxide. After a suitable quantity of water has been run the

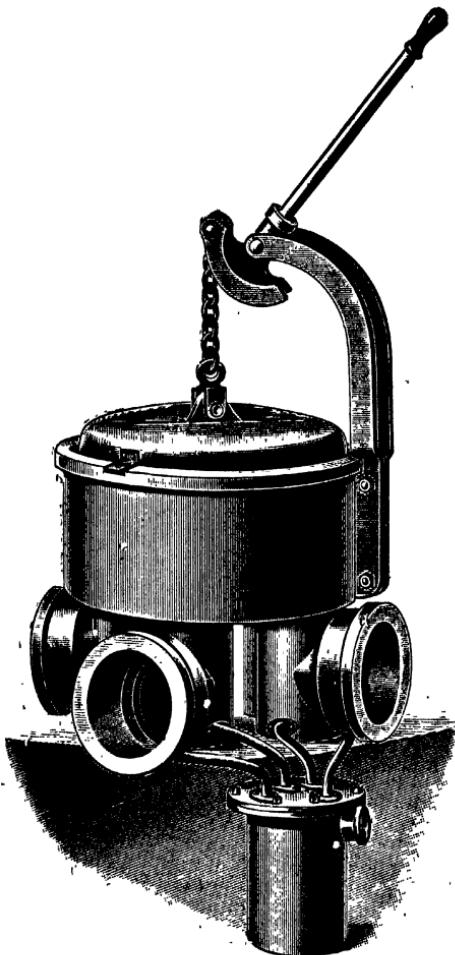


FIG. 96.—Walker Hydraulic Change Valve.

bottles are disconnected and taken to the laboratory, where their contents are emptied into an evaporating dish, a little

## PLANT FOR MANUFACTURE OF SULPHATE

methyl orange solution added, and titrated with caustic soda solution, mentioned in Chapter I., each c.c. of which will represent 0.617 grain. The result divided by the number of feet aspired will give the number of grains of acidity per cubic foot, estimated as equivalent to  $\text{SO}_3$ .

Instead of using the normal solution of caustic soda mentioned above, a special solution of the substance may be made up by dissolving 100 grains of it in water and making up to 1 litre. 1 c.c. of this solution will represent 0.1 grain  $\text{SO}_3$ .

The last method of dealing with the waste gases, and that which is perhaps most suitable for works of small capacity, is that of passing them through the familiar purifiers charged with oxide of iron in a similar manner to that adopted with illuminating gas. As most of our readers will be more or less intimately acquainted with their working in the gasworks, a description of them would be superfluous.

To prevent accidents when changing from one purifying box to another, it is necessary that a suitable valve should be used. Such a one, made by Messrs C. & W. Walker, Limited, is shown in Fig. 96.

The waste gases are of a highly poisonous nature, and whenever there is any chance of a man breathing them (as in repairing a leakage or cleaning out a portion of the plant dealing with them) a suitable respirator or helmet, such as will be described in Chapter XI., should be worn. The method of dealing with a man affected by these gases is also described in a printed sheet mentioned in the same chapter.

## CHAPTER XI

### STARTING, WORKING AND STOPPING THE PLANT—DIFFICULTIES IN WORKING AND THEIR REMEDIES

At a convenient spot, as near the saturator as possible, should be fixed a small bench or broad shelf, and on this should be arranged the following articles:—

Two lead cups, each about 2 inches diameter and the full height of the hydrometers used. The first, which should have a handle similar to that shown in Fig. 97, is used for testing the strength of the sulphuric acid. The second may be similar and have a stout piece of copper wire attached to the handle, or it may have a long lead handle similar to that in Fig. 98. This is used for dipping into the hot mother liquor in the saturator to secure a sample for testing.

A single burette stand and 50 c.c. burette. (The bottom of the latter should have india-rubber tube and pinchcock, not a glass tap.)

A 1 c.c. pipette.

A porcelain evaporating dish about 4 inches in diameter.

Glass rod for stirring.

A wash bottle.

A Winchester quart bottle full of caustic soda, solution containing 8.16 grammes per litre.

A bottle of methyl orange solution.

A Twaddel hydrometer No. 6 and one No. 7 for testing the strength of the sulphuric acid.

A Twaddel hydrometer No. 3 for testing the strength of the mother liquor.

A book of red litmus papers (the kind on paper like writing-paper is the best; those on porous paper like blotting-paper being very bad to see).

*Starting and working the plant.*—And now, having carefully examined our plant to make sure that everything is in its place, we are prepared to make a start.

First, ascertain that all valves or cocks on the waste gas pipes are open and the seal pots on this portion of the plant efficiently sealed with water.

Draw out the small wood plugs shown on the saturator in Figs. 47 and 48, *g* and *k* (pages 59, 60). Place them where they will be readily found when required a little later on.

## STARTING, WORKING, AND STOPPING PLANT 103

Now run Sulphuric acid from the overhead tank to the saturator until the level is about 2 inches over the top of the horizontal portion of the ammonia pipe; then stop the supply.

Run or pump ammoniacal liquor into the still through the superheater until it just shows in the water-gauge glass in the liming chamber, then stop it.

Now pump *water* into the still by means of the lime pump until it shows about 3 inches or 4 inches deep in the water-gauge glass in the bottom chamber of the still, then cease.

Steam is now turned on very gently in the bottom chamber of the still, and as the still gradually warms up, the steam is slowly increased. To prevent any excessive pressure in the still, a suitable reducing valve is inserted in the steam pipe leading to the still, the outlet of which is regulated to about 20 lbs. pressure.

While this is going on the charge of lime is put in the lime mixer, so as to be in readiness when required. The quantity of lime required will vary somewhat according to the composition of the ammoniacal liquor, but will range approximately from about  $2\frac{1}{2}$  cwts. to  $3\frac{1}{2}$  cwts. per ton of sulphate of ammonia produced. Thus a plant producing 4 tons sulphate of ammonia per twenty-four hours would require about 12 cwts. of lime during that period, fed in in small quantities at the rate of  $\frac{1}{2}$  cwt. per hour. The pump should run as slowly as possible, and the quantity of water or waste liquor used to slake the lime should be just enough to keep the pump steadily and continuously going.

Meanwhile the heat will have been steadily travelling up

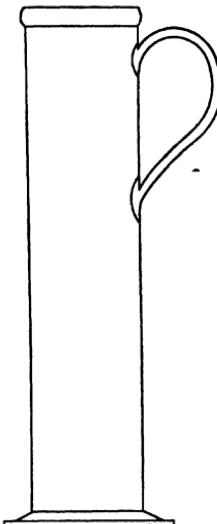


FIG. 97.

Lead Cups.

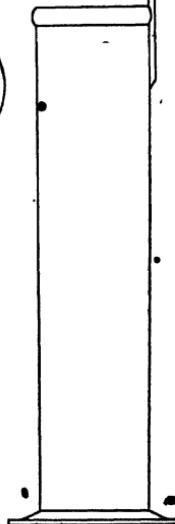


FIG. 98.

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the still, and after a time a little steam will issue from the plug hole in the ammonia pipe in the saturator. Now insert the wood plug, tapping it firmly home. It will now take some little time to heat up the acid contents of the saturator, but as soon as this is done steam will issue from the plug-hole in the waste gas pipe. When this takes place insert the plug and carefully watch the superheater. As soon as heat begins to leave it, turn on a little water to the condensers, regulating the quantity so as to keep the outlet gases quite cool. As soon as the superheater is hot to the touch a little ammoniacal liquor may be turned on, the lime pump started pumping milk of lime into the still, all the steam turned on to the bottom chamber of the still and a little in the liming chamber. The quantity of ammoniacal liquor may now be gradually increased until the full quantity is reached, taking care that it is not done so rapidly as to cool the superheater.

After the still has been working a short time a sample of the spent liquor may be drawn from either the sampling cock or the bottom water-gauge cock, the most suitable thing to draw it into being an iron saucepan about 4 inches in diameter. This waste liquor should be quite milky in appearance and should not smell of ammonia. If it does smell of ammonia it shows that there is either too much liquor passing down the still or that there is insufficient steam on. The remedy is obvious. Next a little powdered lime is placed in the saucepan (a supply of this should be placed in a box in a convenient position) and a second sample of waste liquor run into it. If it did not smell of ammonia in the first test but now does so (and especially if the first sample did not appear sufficiently milky) it indicates that there is not enough milk of lime passing into the still. The remedy is again obvious. These tests should be made at frequent intervals. For more accurate tests of the spent liquor the reader is referred to the next chapter.

We now return to the saturator. As the contents get hotter the motion increases, until, in the case of saturators of open pattern, there is quite a violent swish from side to side, and as a considerable amount of condensation takes place in starting up, it is not unlikely that the contents increase so much in volume that there is danger of them splashing out. To prevent this, sufficient is baled out on to the nearest draining floor or into a mother liquor tank, and this is afterwards used up with the other mother liquor. From time to time the plug is withdrawn from the waste gas pipe momentarily, and a moistened red litmus paper held in the steam, care being taken to avoid breathing this gas, as it is very poisonous. If the litmus paper turns blue it indicates that there is not enough acid in the saturator and some more must be added at once. A test taken of the

## STARTING, WORKING, AND STOPPING PLANT

contents of the saturator at starting would necessarily be the Tw. of the acid, but as the ammonia comes into it, the Tw. gradually decreases, and when it reaches the neighbourhood of  $60^{\circ}$  Tw. the motion in the contents of the saturator gradually steadies, a crystalline skin forms on the surface, and the sulphate of ammonia begins to drop to the bottom. As soon as the quantity warrants it, it is removed either by hand fishing, steam ejector, or bottom discharge valve to the draining table.

A thin stream of acid is turned on. The regulation of this is the most difficult portion of the learner's task, but a little experience soon makes it quite an easy matter. The simplest method of regulating is by means of the hydrometer. By experiment it is found that a particular strength suits the individual plant (in one works the writer found that  $54^{\circ}$  Tw. to  $56^{\circ}$  Tw. suited well, while in another plant  $59^{\circ}$  Tw. to  $61^{\circ}$  Tw. was the most suitable range). An increase in the acid increases the density and *vice versa*.

The most satisfactory way of regulating the supply of acid is by taking a sample of the contents of the saturator and titrating it for free acid, and we have found that an intelligent workman can easily do this. From the sample cup 1 c.c. is rapidly taken by means of the pipette and blown out into the evaporating dish, diluted with water and a little methyl orange added. It is then titrated with the caustic soda solution mentioned at the beginning of this chapter and the number of c.c.'s taken read off. 1 c.c. = 1% of free sulphuric acid. If the contents of the saturator are kept between 3% and 8% very little trouble will be experienced.

If the contents of the saturator get short of acid a test with the litmus paper of the waste gases will immediately show it. In very bad cases the salt assumes a blackish appearance as if mixed with blacklead, while, on the other hand, if the salt is too acid it assumes a peculiar shiny appearance, which soon becomes easily recognised. This latter salt does not dry nearly so quickly on the draining table as the normal quality.

We must now consider the method of dealing with the mother liquor that drains from the sulphate of ammonia as it is drying. It is collected in tanks and elevated either by means of steam injectors made of regulus metal, or acid elevators similar to those shown in Figs. 30 and 31 (page 44), to the overhead supply tank near the saturator.

Two methods of supplying it to the saturator are available, the first in a series of charges, filling up the saturator as full as may be conveniently done, adding a little extra acid and then stopping the supply of mother liquor entirely. This stops the making for a short time, gives the man in charge a little time for attending to other duties—getting his meals or having

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a rest. When ~~running is resumed the charge is gradually worked~~ down until it is as low as it is safe to go without ammonia escaping, and the process is then repeated. The writer does not recommend this method, however, as it leads to irregularity in working, and he strongly recommends the alternative method of running in a small stream continuously.

In addition to the mother liquor that drains from the sulphate of ammonia there is that produced by the melting up of any dirty sulphate that is removed from time to time from that portion of the drainage floors between the loose surface boards and the leadwork. This is most easily melted by placing it in a small wooden tank, lead lined, covering it with water and turning on a small steam jet. (Care should be taken that this jet does not play directly on to the lead, or a hole speedily appears.) After cooling, the solution is syphoned over into a mother liquor tank and thus augments the supply.

If the supply of mother liquor runs short a little may quickly be made by washing down one of the drainage floors with water from a hose pipe.

In saturators provided with a steam pipe such as is shown in Figs. 55 and 56, the steam should be turned on at least once a shift for a few minutes, as this is a great aid in keeping the ammonia pipes clear, and not only produces better working but increases the life of the pipe materially, as a choke in the ammonia pipe is a fruitful source of splitting along the line of holes.

In order to secure steady and satisfactory working, it is necessary that the heat of the ammoniacal liquor leaving the heater for the still should be constant, and in order to watch this it is most desirable that a thermometer should be inserted in the pipe leading from the heater to the top chamber of the still. This pipe is drilled and tapped and a small iron well-shaped socket is screwed in as shown in Figs. 99 and 100. In this well a little mercury is placed and a metal-cased thermometer, as shown in Fig. 99, is inserted. Instead of this metal-cased thermometer an ordinary glass tube thermometer may be inserted in the well through an india-rubber stopper, as shown in Fig. 100, but with this form there is a much greater liability to breakage.

The ammoniacal liquor should be maintained at a temperature of about 200° Fahr., but the exact figure is not nearly so important as that it should be constant.

If the method of intermittent charging of the mother liquor to the saturator be adopted, it will be found that there will be a considerable drop in the temperature referred to above just after charging, and this drop in temperature generally causes a rather quicker flow of the ammoniacal liquor, bringing about

## STARTING, WORKING, AND STOPPING PLANT 107

a "high" waste liquor, owing to the quantity of steam not being sufficient to cope with the altered conditions. The remedy is to ease off the supply of ammoniacal liquor for a few minutes until the temperature again reaches the normal, when the full supply is resumed.

On the other hand, a stoppage or partial stoppage in the flow of the ammoniacal liquor quickly shows itself by a rise on the thermometer, and enables the fault to be speedily rectified.

Variation in the steam pressure at the boilers will frequently cause a variation in pressure on the outlet side of the reducing valve; it is therefore very desirable that a suitable steam pressure gauge should be placed on the reduced side in such a position that the man at the saturator or still can readily see it, as it is obvious that such variation will affect the working.

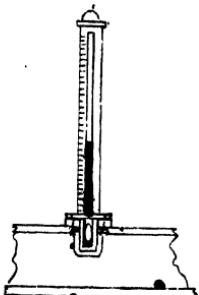


FIG. 99.

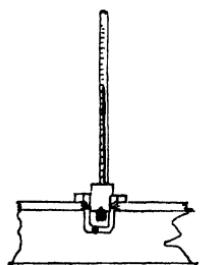


FIG. 100.  
Thermometer Arrangements.

and will require an increase of steam or a reduction in the quantity of ammoniacal liquor to compensate for it.

A very interesting article dealing with these temperatures was contributed by Mr Frederick Shewring to *The Gas World* of 5th December 1914, and to this article the reader is referred for further information on the subject.

After a stoppage of the plant the restarting is effected in a similar manner to that given above, except that the saturator will be already charged, and the still and seal pots already luted. As the charge in the saturator is now mother liquor it will be desirable to use the saturator steam pipe freely on starting the plant, in order to remove any crystals from the ammonia pipe.

*Stopping the plant.*—Work down the charge in the saturator as low as possible, run in about 3 inches of acid, a little water, and turn off the steam.

Stop the ammoniacal liquor. Continue to pump milk of

lime into the still for about a quarter of an hour after the liquor is stopped. The slaking of the lime will have been so arranged that the liming apparatus is now empty.

Keep the steam on for about half an hour after the ammoniacal liquor is stopped and then shut off.

Draw the little wooden plug from the ammonia pipe into the saturator, and open the top taps of the water gauges on the still. This prevents the still, on cooling, syphoning back the contents of the saturator.

Now pump water through the still for about half an hour, through the lime pump, then stop the pump and the plant may safely be left.

If the stoppage is likely to be of long duration it is desirable to empty the saturator into one of the mother liquor tanks and to thoroughly clean out the saturator. A valve is placed on the waste gas pipe in the neighbourhood of the superheater, and care must be taken to always close this before entering the saturator, to prevent any waste gases poisoning the men.

*Some difficulties experienced in working.*—The following are some of the difficulties that may arise from time to time in the working of the plant and the remedies that may conveniently be applied.

*On testing the waste gases at the plug hole, the litmus paper turns blue through the presence of ammonia.*

(a) The contents of the saturator are very low. Run some mother liquor in as quickly as possible and test again. The escape has ceased. Inference obvious.

(b) The contents of the saturator are of normal depth. On testing with the hydrometer, the strength is low, or on testing by titration the percentage of free acid is below 3. Add more acid quickly and test again. The escape has ceased. Inference, again obvious.

(c) The contents of the saturator are of normal depth and the acid is present in sufficient quantity. The escape may stop for a short time if the depth of the charge is high and will probably increase in quantity as the charge works down. The cause is then probably one of two things:—(1) "Hogging," *i.e.*, the building up of hard crystals upon the ammonia pipe until they reach above the surface of the liquor, then a blowhole forms through this mass of crystals, allowing the ammonia to escape without coming in contact with the acid, or (2) a split has formed in the ammonia pipe—generally along the line of the holes—causing a strong *local* blow that the acid cannot effectually cope with.

To ascertain which of these causes is the one a very useful tool for use in open saturators is shown in Fig. 10x. This is made of iron. The flat portion is pushed under the midfeather,

## STARTING, WORKING, AND STOPPING PLANT 109

and passed along the top of the ammonia pipe, when any obstruction due to "hogging" is readily felt. If the pipe feels quite clear of hard crystals and there is a very strong local blow, while there is scarcely any blow at all in other parts of the pipe, there is, in all probability, a split in the pipe.

If "hogging" is found, work down the charge in the saturator as low as possible. Then run in about 3 inches or 4 inches of acid and almost as much water. This will stop the making for a short time and give a heavy boil, which will generally dissolve the hard crystals. A good test to see if the mother liquor is in suitable condition for accomplishing this is to dip in a shovel. If sufficient acid and water be present effervescence (due to liberation of hydrogen) will take place on the metal. If this method does not result in removing the obstruction, repeat, and if it still fails there is no alternative but stopping the plant, emptying the saturator and cleaning it out.

In the event of the pipe being split the only thing to be done is to stop the plant, empty and clean out the saturator, and send for the plumber to effect the necessary repairs. Any attempt at makeshift repairs will be unsatisfactory and will only lead to further trouble and loss of ammonia.

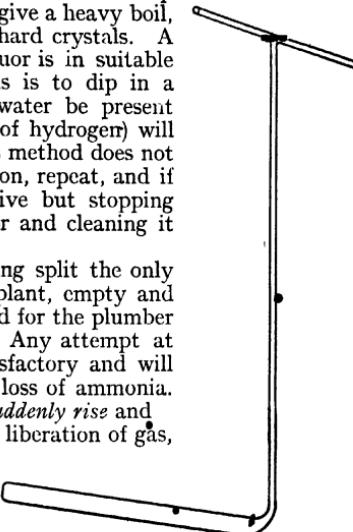
*The contents of the saturator suddenly rise and*

(a) There is a sudden violent liberation of gas, after which the contents drop to the normal depth again, the process being repeated after a short interval.

This is probably due to a stoppage in the condensing plant for the waste gases. On drawing the plug out of the waste gas pipe the steam rushes out violently, while at the termination of the plant (say at the spent oxide burners) very little gas is coming. If suitable small taps have been provided on different portions of the condensing plant, by affixing a U gauge to them and opening them, the position of the obstruction is readily indicated by the difference in pressure. It will usually be found to be due to the choking of a seal pot leading to a lock of liquid in some low portion of the system, and is most frequently found towards the cold end of the plant. It can often be removed by taking off the small flange from the top of the down pipe into the seal pot and probing through the pipe with a stick.

If the obstruction is caused by the choking up of the smaller tubes into the oxide burners it will generally come on more

FIG. 101.—"Hogging" Tool.



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gradually and will not be of so violent a nature, and will have been noticed by the gradually-increasing pressure at the saturator waste gas pipe plug hole.

When opening any portion of the waste gas system it is desirable to use respirators, such as are described in the next chapter.

(b) The contents continue to rise, unless we stop the supply of ammoniacal liquor to the still, and do not drop to the normal depth again. This "boiling over" is accompanied by a characteristic sour and disagreeable smell which is readily recognised. This generally occurs shortly after starting up the plant for the first time, and is then due to some error in fitting up the still, or after the plant has been working for some weeks, and is then due to the choking up of the still with lime compounds. It may also be caused by carelessness, in pumping tar into the still. The only remedy is to stop the plant, in the former case to make good the defect and in the latter cases to clean out the still.

*Discoloration, etc., of the sulphate of ammonia.*—The salt is *orange coloured*, and fumes of the same colour rise from the sulphuric acid as it runs into the saturator. The fault is due to too many nitrous compounds having been left in the acid. Communicate with the manufacturer of the acid, who will only be too pleased to endeavour to put the matter right, as to send away nitrous compounds is a serious loss to him.

The salt is *brown*. This may be due to the presence of an excessive amount of arsenic in the acid, especially if it is made from pyrites. The salt is *dark grey*, as if it had been mixed with blacklead. This suggests that the saturator has been short of acid, as already explained. The salt is *blue*, especially after drying. A fruitful cause of this is the stopping up of the small syphon pipe from the baffle box to the still, thus allowing a little raw liquor to work over to the saturator and form the prussian blue there. To ascertain if this is the case feel the pipe in question; if it is cold it suggests that it is stopped up and must be cleared. The most fruitful cause, however, is the "boiling over" of the still as it is getting choked up and requires cleaning out. Some years ago the Alkali Inspector mentioned a case where it was caused by an iron stay in a saturator having had its lead coverings eaten away. This interesting subject is discussed in several recent annual reports of the Chief Inspector under the Alkali Works Act, and our readers are referred to these for further information.

*Wet sulphate.*—When the sulphate is dried by simply allowing it to drain on the warehouse floor the bottom few inches will generally be found to be more or less damp. This is technically known as "bottoms," and as it is not fit to pack it is disposed of by throwing it on the top of the freshly-made salt. If it gets

## STARTING, WORKING, AND STOPPING PLANT 111

cessive in quantity it is a sign that the space between the loose floor boards and the lead is getting choked up with crystals. The loose floor must then be taken up and the crystals removed, after which they may be dissolved up to make mother liquor, as previously described.

It occasionally happens that there are portions of the sulphate of ammonia (especially the lumps) that will not dry, even if kept for weeks, and if a small portion of this salt is broken up in the hand it feels quite sloppy. It is also strongly acid. If the saturator has not been worked in an excessively acid condition this sloppiness is generally due to impurities in the sulphuric acid, especially if some acid has been used that has been used for washing down a Glover tower, and complaint should be made to the manufacturer.

The writer would, however, point out that there is a strong tendency on the part of the saturator men to blame the acid whenever anything goes wrong with the working, and from long experience in making both sulphuric acid and sulphate of ammonia he has found that in the majority of cases the acid maker is not at fault, and a little further investigation would prevent many complaints being made.

## CHAPTER XII

### COST OF MANUFACTURE OF SULPHATE OF AMMONIA

SINCE<sup>111</sup> the first edition of this work was issued, the publishers and the writer have had several inquiries as to the cost of manufacturing sulphate of ammonia.

This is a somewhat difficult question to answer, as so much depends upon the cost of carriage, labour, etc., that scarcely any two works are alike. While the writer cannot give figures in £, s. d. that could be taken as a useful guide, he purposed in this chapter to discuss the matter in such a manner that it will enable the reader, with his knowledge of the local conditions, readily to get out the information for himself.

The cost of production is best dealt with under the following heads, viz. :—

#### Materials required :—

Ammoniacal liquor.

Sulphuric acid.

Lime.

Fuel.

Bags for packing the sulphate of ammonia.

#### Wages :—

Manufacturing account.

Repairs account.

Material for repairs.

#### General Expenses :—

Interest on capital.

Rent, rates and taxes.

Management, clerical work, etc.

Depreciation of plant.

Carriage of the sulphate of ammonia.

## MATERIALS

*Ammoniacal Liquor.*—The method of ascertaining the number of tons roughly required to make a ton of sulphate of ammonia, is explained fully in this work on page 136, Chapter XV. The value of the liquor may be taken as that of one's existing contract or may be taken from the quotation in the current numbers.

of the trade journals, due allowance being made for any saving of carriage due to using up the liquor in one's own works.

In some works it is the custom to debit all the other expenses first, and then take the difference between that and the value of the sulphate of ammonia as being the value of the ammoniacal liquor, the difference between the enhanced value and the price obtainable by selling as liquor representing the profit through converting into sulphate of ammonia.

*Sulphuric Acid.*—As mentioned elsewhere (page 136, Chapter XV.) 1 ton of sulphate of ammonia requires about 14.84 cwts. of the pure acid (= 18.32 cwts. at 144° Tw.) for its manufacture. The prices will be obtained from the nearest manufacturers. If the reader is a manufacturer of sulphuric acid, we need scarcely point out the saving of carriage effected by using it on the works instead of delivering to customers, also the reduction in cost of production of the acid due to utilising the sulphuretted hydrogen in the waste gases from the sulphate of ammonia process as a portion of the source of the sulphur for the acid manufacture.

*Lime.*—The quantity of lime required will vary with the different ammoniacal liquors. Those containing the greater proportion of non-volatile ammonias will, of course, take most lime.

At one works with which the writer was connected for nine years, the quantity required during those years averaged 2.95 cwts. of lime per ton of sulphate of ammonia produced. In another works with which he was intimately acquainted, the average during five half-years was 3.12 cwts.

Only lime of first-class quality should be used, such as the limes of the Buxton, Skipton, Silverdale, etc., districts; lime of inferior quality has a tendency to choke up the stills.

It is obvious that here again carriage has much to do with the cost.

*Fuel.*—During the nine years at the works mentioned above the fuel averaged 20.39 cwts. per ton of sulphate of ammonia produced, but (except during the first year) the bulk of this fuel was only fine coke dust after all the better qualities had been riddled out. It was augmented by coal only when supplies of coke dust ran out.

At the other works the average during the five half-years was 10.77 cwts. per ton, but here coke of good quality was the fuel.

Unfortunately the writer has not figures available where coal is used as fuel.

*Bags for the Sulphate of Ammonia.*—They bags usually used are second-hand meat or sugar bags, the former generally being the best. Some works fill just 2 cwts. net, in each bag,

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while others fill to the full capacity of the bag. String for sewing the bags has also to be allowed for. Where double bags are required, the buyer usually pays extra cost.

### WAGES

Here again local conditions are very different. At one of the works referred to above the following men were employed when making about 90 tons of sulphate of ammonia per week:—

Foreman (about two-thirds of his time on sulphate of ammonia; remainder on sulphuric acid process).

Three boiler firers (each on 8 hours' shift).

Six saturator men (each on 8 hours' shift).

Two lime pump youths (each on 12 hours' shift).

About seven or eight labourers on day only. The labourers would also assist at times in other departments.

During the nine years the cost per ton of sulphate of ammonia produced averaged 9s. 2d. for process account, and 1s. 4d. for repairs. It should be added that at the commencement the plant was new and cost nothing in repairs during the first three and a half years. At the other works the cost for the five half-years averaged 6s. 2½d. per ton for process account, and 4d. per ton for repairs.

I should say that it is a few years since these figures were taken, and since then wages have been considerably increased, especially in the town where the lower figures prevailed.

### MATERIALS FOR REPAIRS

The writer has no figures available for this, but the cost (unless the plant is remodelled) is not heavy. A plate lead saturator should last about twelve years and at the end of that time should yield about two-thirds of the original lead as scrap lead.

### GENERAL EXPENSES

*Management.*—In the works in which the writer spent nine years as superintendent or works manager he had a young man as assistant. Between us we took the management of the whole chemical works, including the sulphuric acid plant, drew most of our plans and did the whole of our laboratory work, kept the process books and furnished the necessary reports to the chief office.

The other chemical work was done jointly for the chemical works and adjoining gasworks, while the accounts were kept at the chief offices, in the neighbouring city.

At a small works he knows, the management is undertaken

by the gas manager and coke ovens manager jointly, both these undertakings and others belonging to the same firm. The laboratory work is done by a young man assistant, whose time is only partly occupied in this manner.

Readers will understand the other items of general expenses quite as well as the writer.

#### CARRIAGE OF THE SULPHATE OF AMMONIA

Where the works are situated in the centre of an agricultural district, much of the sulphate of ammonia may be sold locally, but otherwise a considerable quantity is exported through the ports of London, Liverpool, Hull, Gool<sup>ee</sup> and Leith.

In addition to the cost of the carriage to one of these ports, there will be the shipper's charges for placing the sulphate f.o.b. (loading, sampling, re-weighing, etc.), also the broker's commission (usually 1%) for selling.

## CHAPTER XIII

### MANUFACTURE OF CRUDE AMMONIA OR CONCENTRATED AMMONIACAL LIQUOR

SINCE the issue of the first edition of this work the publishers and the writer have been frequently asked to furnish similar particulars of other branches of ammonia manufactures. While we should like to be able to supply the manufacturing public with the information they desire, there are many reasons that prevent our being able to do so.

The market for sulphate of ammonia is comparatively unlimited, the manufacturing process is a fairly simple one,

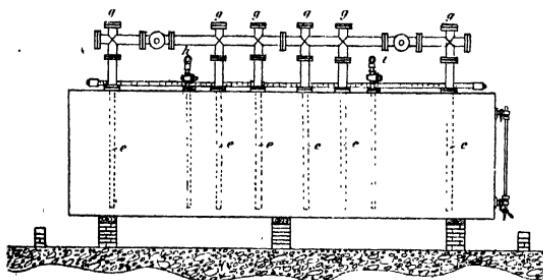


FIG. 102.—Crude Ammonia Plant. Front Elevation.

and the manufacturers are most willing to assist each other in it. On the other hand, the market for many of the other products is a very restricted one and unlikely to expand, the processes are far more complicated, and in many cases the best processes are of a secret nature, so that the writer feels that it would be unkind to his old friends to reveal them.

While this applies to many of the processes, there is, however, one process that he feels at liberty to give particulars of, viz.—the manufacture of crude ammonia, or, as it is sometimes named, concentrated ammoniacal liquor.

Under certain exceptional circumstances, this process might

be a useful alternative to sulphate of ammonia manufacture, but the writer would warn the reader at the very outset that the process is not as simple as it appears, and that many difficulties will present themselves that can only be overcome by patient experimental work in each individual case, if it is intended to obtain anything like good results.

The plant is shown in Figs. 102, 103, and 104. A concrete foundation is put in, and upon this a low boundary wall about 14 inches high is built of bricks laid in Portland cement, the

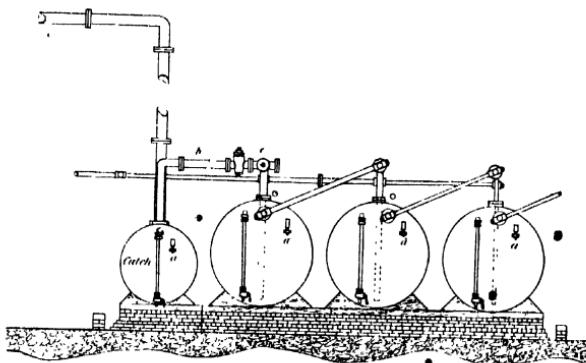


FIG. 103.—Crude Ammonia Plant. End Elevation.

whole forming a tank to collect the water which is used for cooling the receivers, and passing it away either to the drain or to some place where its heat may be utilised.

The plant itself consists of the still, liming arrangements, etc., as in sulphate of ammonia manufacture, but instead of a saturator there are four or more boilers, known as the "Catch" crude receivers, Nos. 1, 2, 3, etc. (for convenience generally spoken of as C.R. 1, 2, 3, etc.).

In works where a sulphate of ammonia plant is in existence, it is desirable to make the connection from the still in such a manner that by turning the tee pipe at the top of the still round it may be made to serve for either the sulphate of ammonia plant or crude ammonia plant as desired. This arrangement is shown in Fig. 105. The pipe end not in use should be closed with a blank flange to prevent smell.

The delivery pipe from the still leads to the "catch," and in this case does not enter the boiler as a "dip pipe."

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The "catch" and the receivers are made from old steam boilers, either the old-fashioned egg-ended or dish-ended types, or old Cornish or Lancashire boilers from which the tubes have been removed, and the ends suitably plated up. As the pressure

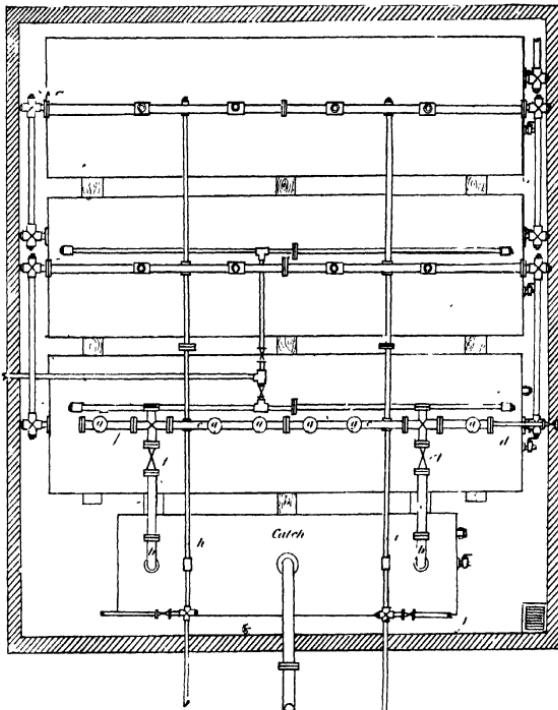


FIG. 104.—Crude Ammonia Plant. Plan.

scarcely exceeds 10 lbs. per square inch, it is obvious that no great strength is required.

The "catch" and each receiver is fitted with a suitable water gauge made of *malleable iron* (brass will not be found at all suitable). Each is also fitted with a "vacuum valve" made of cast-iron and shown at *a* in Fig. 103, and in full detail in Figs. 106, 107, 108, and 109. To attach these vacuum valves, a hole is cut in the end of the boiler, tapped, a nipple inserted, a cast-

iron cock is attached, and to this is fastened another nipple and a tee, as shown in Fig. 110. The vacuum valve is inserted in the lower branch of the tee. While working, the pressure inside the catch or receiver keeps the valve closed, but when stopping the plant or pumping from it, vacuum forms, the mushroom of the valve lifts, and prevents any syphoning over of the contents of the receivers. When the vacuum is satisfied the mushroom drops and prevents any escape of ammonia. In the upper branch of the tee it is desirable to place a pressure gauge. An even better arrangement is to reverse the position

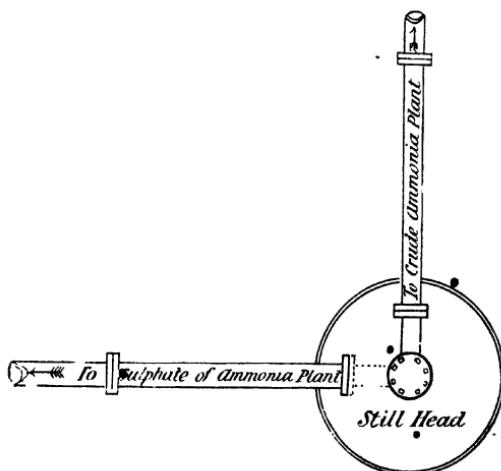


FIG. 105.—Crude Ammonia Plant. Connection from Still Head.

of the tee, put the vacuum valve in the lower branch, and attach a syphon pipe to the horizontal one, to which is then connected the pressure gauge. By filling this syphon pipe with oil the life of the pressure gauge is considerably lengthened. It is desirable to remove, examine, clean, and oil the vacuum valves at frequent intervals, as there is a considerable tendency for them to rust or crystallise up; hence the necessity for the cast-iron cock shown.

The use of the "catch" is to retain the usually weak liquor caused by the rapid condensation of the steam that comes from the still, and thus enable the much drier gas to pass forward to the receivers. The weak liquor thus formed may be used for charging up the first C.R., but if more is formed than is

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required for this purpose the best way is to return the 'excess to the ammoniacal liquor store for redistillation.

From the "catch" the gases pass along the two pipes *b*, *b*, in Figs. 103 and 104, to the main, *c*, along the top of the first receiver. In each branch a cast-iron cock, *f*, is placed, so that they can be closed against the still, and steam turned on in the pipe, *d*, shown in Fig. 104, to clear away any choke due to crystallisation. From the main pipe the gases are led into the receiver through a series of about six "dip pipes," *e*. These dip pipes are best made of light cast-iron similar to the pipes used for the heating of greenhouses, but having a flange at one end.

Fig. 108.

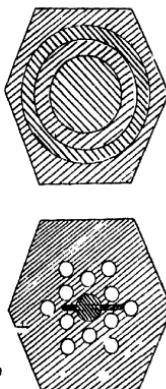


FIG. 106.

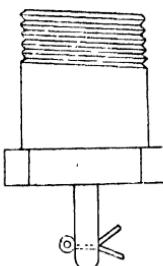


FIG. 107.

Vacuum Valve.

FIG. 109.

The main is of cast-iron with flanges, *g*, over each dip pipe, so that they can easily be removed to clear the pipes from crystals.

From No. 1 C.R. the gases pass along wrought-iron pipes to the main and dip pipes of No. 2 C.R. These also are of wrought-iron, a plug in the top of the cross taking the place of the flange for probing purposes. The arrangements from No. 2 C.R. to No. 3 C.R. are again similar to the last-mentioned.

From the last-named receiver the gases pass away either to the oxide burners, or Claus process, as shown in the sulphate of ammonia plant, or they may be sent through an oxide of iron purifier. As the quantity is very small, it is obvious that the box need not be of large capacity.

A suction pipe, *h*, Figs. 102 and 104, passes over the whole series, and has a branch with a tap into the catch and each receiver. There is also a branch on the same series connected

with a suitable water supply, the whole being connected to a suitable pump. The delivery pipe from the same pump passes over the whole series, as shown at *i*, in the same Figs., and this has a branch, *j*, to the store tanks. Another branch should be provided to take the weak liquor back to the ammoniacal liquor store. It is obvious that such an arrangement enables water to be pumped into any of the receivers and the contents of either catch or any receiver to be transferred to any other of the series. Provision must also be made for getting the crude ammonia from the store to the railway tanks. If the stores are sufficiently high they will fill the railway tanks by gravitation, but if not, suitable pumping arrangements will

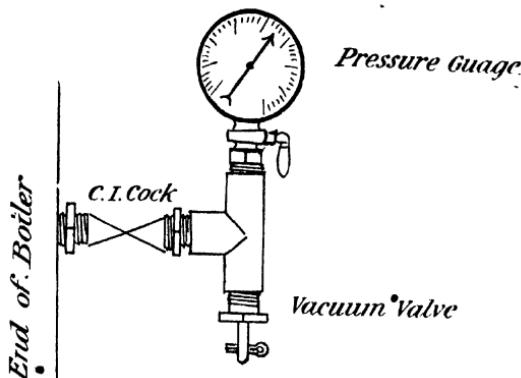


FIG. 110.—Pressure Gauge.

require to be made. It is best to have a separate pump for this purpose, as it may happen that at the time it is desired to load a railway tank the receiver pump may not be available.

2-inch wrought iron pipes perforated with small holes pass along the tops of receivers, Nos. 1 and 2. These are supplied with water either by means of a pump or an overhead tank, and are used for cooling the receivers.

The wear and tear on the catch is very heavy. The writer's experience is that they rarely last more than eighteen months to two years. C.R. 1. generally lasts about four years, while the other two last much longer.

To start the plant, each of the three receivers has water pumped in until the dip pipes are just nicely sealed. The still is then started in exactly the same manner as in sulphate of

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ammonia making; but it is desirable to use as slight an excess of steam as possible in order to avoid making weak crude.

Soon the condensings will begin to show in the water gauge of the catch; and when it gets about half full a sample may be drawn and tested. It may be of full strength, *i.e.*, about 15%  $\text{NH}_3$ , in which case it may be pumped straight away to the store tank. It is, however, more likely to be below this strength, in which case, if there is plenty of room, it may be pumped into C.R. No. 1. If there is not room there, the alternative is to pump it back to the ammoniacal liquor store, for re-distillation.

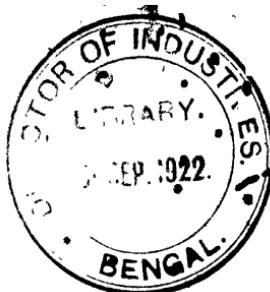
The contents of No. 1 C.R. will gradually increase in strength until they reach about 15%  $\text{NH}_3$ , when they may be pumped away to the store tank. A portion of the contents of No. 2 C.R. is pumped into No. 1 C.R. to re-charge it, a portion of the contents of No. 3 C.R. is pumped to No. 2. C.R., and a little more water is pumped into No. 3 C.R., and thus the process goes on, no stoppage of the plant being necessary while the pumping proceeds.

Tests of the contents of the catch and crude receivers are made once or twice a day, and, according to the results, instructions for pumping are issued. The make is chiefly in No. 1 C.R., No. 2 C.R. rarely gets above 4%  $\text{NH}_3$ , and No. 3 C.R. should not be allowed to get beyond 2%  $\text{NH}_3$ .

The desideratum for crude ammonia for use in the ammonia-soda process is 15% to 18%  $\text{NH}_3$  and as much  $\text{H}_2\text{S}$  as possible, 3% being very acceptable, and the buyers do not like it less than 1½% to 2%. It is sold on the basis of the percentage of ammonia, at so much per unit of  $\text{NH}_3$  per ton, a minimum percentage requiring to be guaranteed.

The main thing is to avoid getting flooded out with weak liquor, and the chief difficulty is the strong tendency for the crude ammonia to crystallise during cold weather.

It may be of interest to note that when the crude ammonia crystallises out, the crystals themselves will be much stronger and the liquid left much weaker in  $\text{NH}_3$  than the original liquid before crystallising.



## CHAPTER XIV

### MANUFACTURE OF SULPHATE OF AMMONIA IN SMALL WORKS

THE plants mentioned in the earlier parts of this work are of sizes adapted to the requirements of works of various sizes, but it may happen that in a very small works no spare steam is available for such a plant, or labour suitable for the continuous process is not conveniently to be found.

To meet such cases Messrs C. & W. Walker, Limited, have devised the small plant shown in Figs. 111 and 112, where A is the still, B the lime mixer, C the saturator, D the condensing plant, E the oxide purifier, and F the store for sulphate of ammonia. This plant is suitable for gasworks producing only a small quantity of ammoniacal liquor, and with limited capacity for storage of liquor.

The apparatus consists of a horizontal still, baffle box, saturator, mother liquor well, draining table, and stores for sulphate, condenser, devil liquor siphon, purifier, and liming tank.

The still consists of a steel boiler 3 feet in diameter by 9 feet long, built in a brick setting, with furnace door and frame, fire-bars, dampers, etc., and steel chimney. The furnace gases pass under the boiler, and return in brick flues along each side to the chimney. The still is fitted with gauge fittings and glass pressure relief pipe, vapour dome, manhole, sludge outlet, lime inlet, and liquor inlet. The "free" ammonia is first driven off, lime added, and the distillation continued until the spent liquor contains the lowest percentage of ammonia desired.

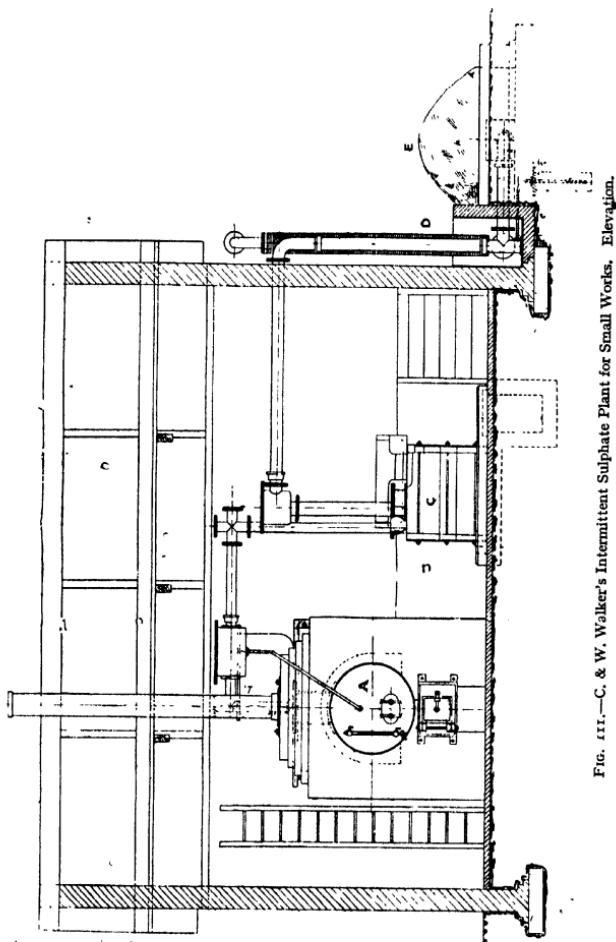
The cast-iron baffle box consists of a  $\square$ -shaped box with diaphragm, and drain pipe for arresting the aqueous vapour carried mechanically forward with the ammonia gas, the drainage being returned to the still.

The liming apparatus consists of one mixing tank with wire screen and draw-off pipe and cock, and is fixed on an elevated platform for running direct into the still.

The saturator is of the usual fishing type, formed of pure chemical lead in a stout timber case with a pressure rated ammonia pipe and waste gas outlet. The saturator is supported on an ordinary brick or concrete foundation, and is placed in a shallow lead tray having a fall towards the mother liquor well.

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The mother liquor well can be formed either in concrete or brickwork, and lined with chemical sheet lead, or can be made



from pitch pine in the form of a tank, and lead lined. The latter has the advantage that any leakage can be easily detected and made good.

## MANUFACTURE OF SULPHATE IN SMALL WORKS 125

The draining table consists of the usual timber lead-lined tray, placed so as to run the drairage back to the saturator, and is made large enough to hold the day's make.

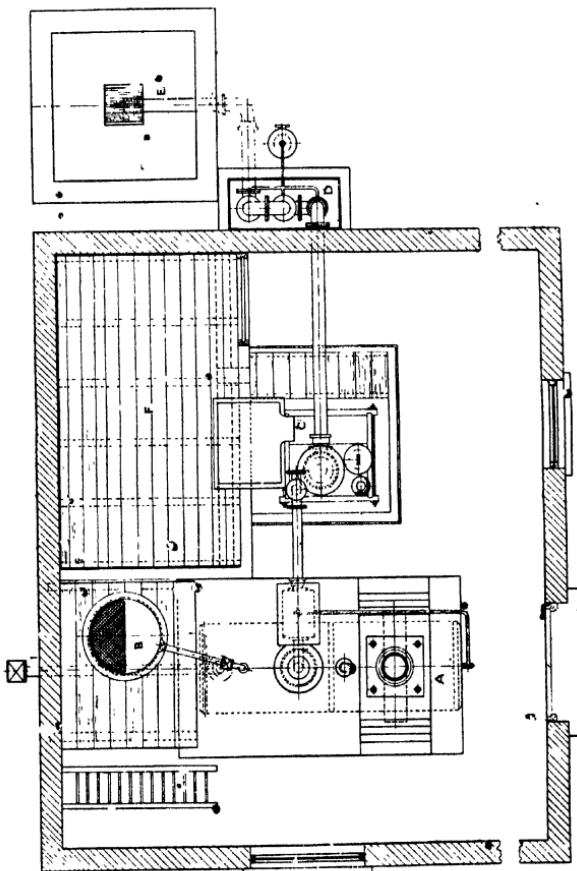


FIG. 112.—C. & W. Walker's Intermittent Sulphate Plant for Small Works. Plan.

The stores for sulphate are large enough to store a 10-ton lot, the bottom being lined with light sheet lead, and protected with loose timber flooring in the usual way.

The condenser consists of vertical pipes, the lower portions being immersed in a water pit. A small devil liquor syphon

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is provided, and a water spray arranged for use in very hot weather, so as to cool the waste gas to the desired temperature.

The purifier consists of a small brick box with the gas inlet pipe carried to the centre, and covered with a wood grid, the oxide being heaped up as required.

This plant is easily worked by unskilled labour, and its small cost allows of a good return for the capital invested.

The following exceedingly interesting paper read before the Southern District Association of Gas Engineers and Managers at their meeting on 5th November 1910 appeared in *The Gas World*, together with a leading article and a report of the discussion which followed the reading of the paper. By the kind permission of Mr Moon and the Editor of *The Gas World*, we have pleasure in reproducing the paper, as follows:—

Ammonia, the residual from which gas undertakings of a fair size expect to make a respectable profit, is to very small works often nothing but a nuisance and a source of loss. Any ammoniacal liquor that may be made presents great difficulty in its disposal, as the cost of carriage absorbs all the profit on its sale, unless the works are very favourably situated; and the capital expenditure necessitated by the installation of a sulphate of ammonia plant of the ordinary type would not in most cases be justified, in view of the small quantity of liquor to be dealt with and the fact that the plant would be standing idle for, say, eleven months during the year. Furthermore, sulphate plant of the ordinary type requires constant attention of a more or less skilled character, and monopolises the whole time of one man while it is in operation.

It is, therefore, not an uncommon thing to find that in a small works no attempt whatever is made to recover ammonia from the gas. The water in the purifier lutes and gasholder tanks is converted in time into weak liquor; and the gas distributed to the consumer is full of free ammonia. The inevitable result is trouble on the district caused by the sticking of taps and the corrosion of burner nipples and fittings. The weak liquor that is unavoidably produced on the works is allowed to drain away, either into some adjacent water-course or into the public sewer, and is bound sooner or later to give trouble.

The works at Wareham, supplying about 5,000,000 cubic feet of gas per annum, were in much the above plight about eighteen months ago. We were concerned as to how best to improve matters; and on the suggestion of Mr Wilton we decided to instal a small experimental plant on what are, I believe, somewhat novel lines—at any rate, in ordinary gas-coal practice. At Wareham there is only one man on shift by day and one by night, with the manager to read meters, do all necessary

fitting work on the district, and attend to public lighting, etc. Obviously any plant to be successful would have to be so simple that an ordinary country workman could not fail to understand it, and so arranged that it would not entail any considerable extra labour on the staff.

The plant installed at Wareham is shown in Fig. 113, and has now for several months proved to fulfil admirably the above conditions! The coal gas, after passing the condensers and a small purifier filled with heather, which acts as a some-

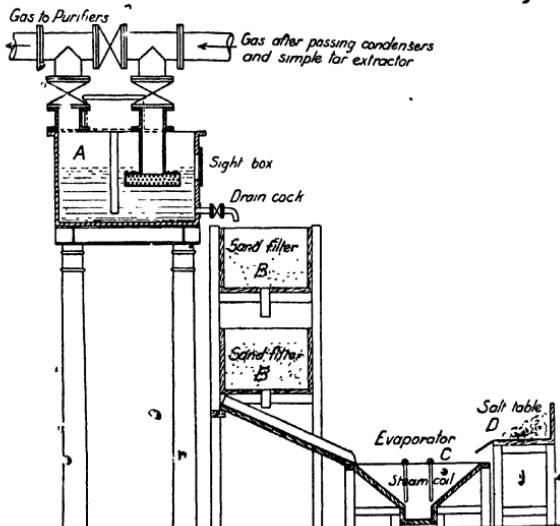


FIG. 113.—Sulphate Plant at Wareham Gasworks.

what crude tar extractor, enters the lead saturator A, and bubbles through the weak sulphuric acid contained therein.

We have proved by careful experiments that there is no apparent loss of illuminating power due to the absorption of hydrocarbons from the gas, so long as the strength of the acid is kept below  $4^{\circ}$  to  $5^{\circ}$  Twaddel. In starting the acid washer, therefore, the acid is mixed to about  $3^{\circ}$  Twaddel. At this strength, the avidity with which it will absorb the ammonia renders it unnecessary to work with more than 1 inch to 1½ inches of seal in the washer. Sulphate of ammonia is formed in solution; and the specific gravity of the washing liquor slowly rises.

A simple calculation gives approximately the amount of

sulphuric acid that should be added from day to day to neutralise the ammonia produced from the amount of coal carbonised per twenty-four hours. This amount is added to the saturator in half quantities each twelve hours. Roughly, about 23 lbs. of acid of 142° Twaddel would be required per ton of coal if all the fixed and the free ammonia had to be neutralised. Where the free ammonia only is dealt with, as at Wareham, about 13 to 16 lbs. only is required. These amounts will, of course, vary according to the kind of coal carbonised and the heats used. Adding acid in this way, in the quantities necessary only for each day's make of ammonia, the specific gravity of the washing liquor increases regularly; but the amount of free acid in solution is never high enough to affect the illuminating power.

When the strength of the solution reaches 45° to 50° Twaddel, it is drawn off in small quantities each day, so as to keep the seal approximately constant, passed through the two small wooden, lead-lined filters BB (filled with sand on a layer of felt), and, when enough has accumulated, transferred to the evaporator C. This, at Wareham, where exhaust steam is available from the exhauster engine, is a wooden, lead-lined vessel of the shape shown, with a flow-and-return lead pipe for the steam. Very little concentrating is needed, as at 45° to 50° Twaddel the sulphate of ammonia solution is very near crystallising point.

In works not using an exhauster, and therefore with no steam available, a cast-iron lead-lined evaporator of convenient shape, sitting either on the waste-gas flue or on top of the beds, would be quite effective. Crystallisation soon takes place, and the salt is put on the draining board D to drain; the small amount of mother liquor left being mixed with the next batch for concentrating. Very good coloured salt is made by this process; and the sample of Wareham salt exhibited gives an analysis of 25.2 per cent. of ammonia. A good local sale can usually be cultivated with very little trouble, as most small works are of necessity situated in a country district.

In a small works such as Wareham, the amount of virgin liquor from the hydraulic main is hardly sufficient to warrant the installing of a lime still to liberate the fixed ammonia. But the whole of this liquor, and the stronger liquor from the condensers, is pumped through the hydraulic main twice; so that as much free ammonia as possible is driven off into the gas. The virgin liquor run to waste at Wareham amounts to approximately 11 gallons per ton of coal, containing free ammonia equal to 1.1 lbs. and fixed ammonia equal to 4 lbs. of sulphate. This is equivalent to a total waste of 5.1 lbs. of sulphate of ammonia per ton. The plant has been working for three months;

and over this period 14 lbs. of sulphate of ammonia have been made per ton of coal carbonised. In addition to this, gas entirely free from ammonia has been distributed, and the waste liquor to be disposed of has been considerably reduced in both strength and quantity.

It is necessary with a process of this kind to keep the saturator liquor constantly on the acid side. If it were allowed to become alkaline, sulphuretted hydrogen and carbonic acid would be absorbed; and when the next acid was added, these gases would be given off in considerable quantities. The purifiers would be overtaxed by the burst of sulphuretted hydrogen, and the illuminating power of the gas would certainly suffer temporarily from the extra carbonic acid, if oxide only were used for purification. Fortunately, it is quite easy to avoid this trouble.

With regard to the financial side of the question at Wareham. As there is no fuel or labour bill to be met, practically the only charges against the sulphate sold are the cost of the acid used and the interest and depreciation on the plant itself. The capital outlay is, however, so small, and the wear and tear of plant so inappreciable, that these charges do not amount to very much per ton. I estimate that, selling the sulphate of ammonia produced at £11 per ton, there will be a net profit of £7 per ton sold. This will yield a total profit for the year equal at Wareham to an increased dividend of 0.6 per cent.

What has been done at Wareham on a very small scale has been done on a larger scale, and therefore much more thoroughly, at Dunstable, by Mr Phillips, who has kindly accorded to me permission to give you the particulars of the plant and his experience with it up to date. At Dunstable, between 2000 and 2500 tons of coal are carbonised per annum; and the fixed ammonia therefore is sufficiently significant to be worth recovery. The gas as it leaves the hydraulic main is passed through a specially constructed condenser-washer, so arranged that, as the liquor condenses, it flows in a reverse direction to the gas, and leaves the apparatus warm and containing very little free ammonia.

This liquor is conveyed to a direct-fired distillation apparatus, where it is treated with alkali and the ammonia driven off to the acid washer through which the main stream of gas passes. The spent liquor, which averages about 10 gallons to the ton of coal carbonised is conveyed to a shallow tank on the top of the retort bench, and is evaporated to dryness. It will be seen that the quantity of effluent, by this system is only about one-fourth of that which would be produced by the ordinary method of water washing and steam distillation. Thus, at a small works carbonising, say, 10 tons of coal per day, there

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would only be 100 gallons of effluent to dispose of ; and this, if desired, could be easily evaporated in the ash-pans.

Referring to Fig. 114, B shows the fractional condenser-washer, consisting of six bubbling trays each working with a seal not exceeding  $\frac{1}{4}$  inch. The hot gases from the hydraulic main enter the washer at the bottom and pass upwards through all the six trays. The two top trays may be used as naphthalene washers by filling them with a suitable solvent, such as oil tar, which also serves to remove the last traces of coal tar from the gas. The middle two trays are provided with cold water circulating pipes, which may be adjusted so that the exit gases are reduced to any desired temperature. The bottom two trays receive the condensed liquor from the trays above, and also the tar and liquor from the hydraulic main. The hot gas bubbling through the liquor in the two trays raises its temperature considerably, drives off most of the free ammonia, and the liquor passes away from the washer at about 100° to 120° Fahr., or very little lower than the temperature of the inlet gas.

Through the exhauster, C, the gas now passes to the acid washer D. This is arranged with trays so that the gas is washed three times. Sulphate of ammonia solution is formed, and is constantly circulated through the receiver Y and overhead tank X. Acid is added at intervals, so that the liquor entering the washer contains 1 per cent. and leaving the washer only 0.1 per cent. of free acid. The strength of the sulphate liquor gradually increases ; and when it reaches 45° to

50° Twaddel, it is run off to the evaporator G, where it is concentrated by means of a small steam coil and crystallized. The

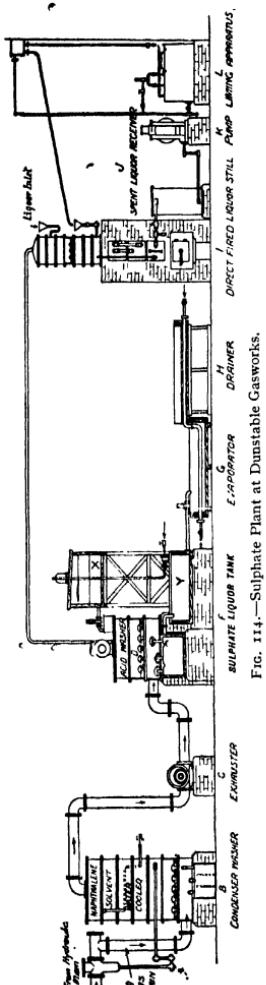


FIG. 114.—Sulphate Plant at Dunstable Gasworks.

saltrainer H is also provided with a steam coil, so that each batch of sulphate can be dried before removal—thus making it unnecessary to have a lead-lined store.

The virgin liquor containing the fixed ammonia is pumped from the outlet of the condenser to an overhead tank, from which it flows by gravity to the direct-fired still I. A direct-fired still is used in preference to the usual type, so as to prevent the increase in bulk of the effluent liquor due to the condensed steam. It is easy to operate; and it is of importance to note that the lime deposit does not adhere as with steam, and is therefore quite easy to remove. Finally, the effluent liquor is pumped into two tanks 8 feet  $\times$  3 feet 9 inches  $\times$  6 inches deep, laid on the top of the retort bench, where it evaporates to dryness.

The whole plant is exceedingly simple to operate, and does not call for any increase whatever in the works staff. Liquor tanks become quite unnecessary, and there are by this process no smells of free ammonia and sulphuretted hydrogen about the works. There is no effluent, and no possibility of nuisance of any kind. It has not yet been possible at Dunstable to obtain figures with regard to the make of sulphate per ton of coal; but as there can be no escape of ammonia at any point in the process the yield must, of necessity, be fairly high. The first plant installed at Dunstable was hardly so complete as that described; but it was sufficiently successful to warrant the construction of the more complete system outlined above.

Engineers who have seen the process in operation at Dunstable have been considerably impressed with its simplicity and efficiency; and at the time of writing this paper, at least four engineers have placed orders for similar plants.

The only criticism that can, I imagine, be made on a process of this kind is that one loses the purifying power of the ammonia for the partial removal of carbonic acid and sulphuretted hydrogen. If the best use possible by ordinary scrubbing methods is made of the ammonia, about 200 to 300 grains of carbonic acid and 100 to 120 grains of sulphuretted hydrogen per 100 cubic feet of gas are removed by the scrubbers. In works using oxide only for purification, the extra sulphuretted hydrogen would be absorbed by the purifiers without trouble, and the carbonic acid would pass forward into the gas, increasing the volume by about 0.5 per cent. and reducing the illuminating power by about 0.3 candle. In most small works this would be quite unnoticed, and would in any case be well within the ordinary variations of illuminating power. Where lime only is used, the net result would be a slight increased use of lime, the cost of which would be much more than covered by the profit from the sulphate, and the great advantage of distributing gas free from ammonia.

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As a matter of fact, in most small works anything like full duty is very seldom obtained from the ammonia as a purifying agent for carbonic acid or sulphuretted hydrogen, and the additional quantities of these gases passing to the purifiers would not amount to anything like the figures given above. Although the process up to the present has been applied to small works only, it has advantages which, under special local conditions, might make it worthy the attention of much larger undertakings. Scrubbers, either of the tower or rotary type, and large liquor storage tanks would become quite unnecessary, and the waste exhaust steam that abounds in many works could do the necessary work instead of the plentiful supply of live steam required by the present methods. In addition to this one would have a practically continuous process of manufacture instead of an intermittent one.

In conclusion, I should like to take this opportunity of thanking Mr Phillips for his kindness in placing the particulars of the Dunstable plant at my disposal, and Mr George Wilton for the help he has given me throughout.

## CHAPTER XV

MISCELLANEOUS: ALKALI, ETC., WORKS REGULATION ACT, 1906  
—FACTORY ACTS (SPECIAL REGULATIONS)—RESPIRATORS—  
SULPHURIC ACID—QUANTITY OF AMMONIACAL LIQUOR RE-  
QUIRED TO MAKE A TON OF SULPHATE—ANALYSIS OF WASTE  
LIQUOR

ALL sulphate of ammonia works are compelled to be registered under this Act. Application for registration (accompanied by a fee of £3) should be made to the Local Government Board, Whitehall, London. The registration requires to be renewed and the same fee paid annually.

This Act is administered in an ideal manner. It has always been the wish of the authorities that the manufacturers should feel that the inspectors under the Act should not be looked upon as police, but rather as the mutual friends of the manufacturers and the outside public, working together for the common good. The inspectors are gentlemen who have a thorough knowledge of chemical works, and during the writer's thirty years' experience of intimate contact with them he has experienced nothing but kindness and consideration at their hands.

Prosecutions under the Act are very rare, and in all cases where they do take place the feeling of the manufacturers is that the delinquents richly deserve the penalties inflicted upon them.

### *Factory Acts. Special Rules for Chemical Works*

A copy of these rules (twelve in number) must be posted up in each chemical works. Copies may be obtained (price, 1½d. post free) from H.M. Stationery Office, Kingsway, London, W.C.2.

Under Rule 9 it is enacted that *Respirators* charged with moist oxide of iron shall be kept for use when required when working amongst sulphured hydrogen or other poisonous gases. Two forms of such respirators, made by Messrs Zimmer & Co., are shown in Figs. 115 and 116. When received, the cavity is filled with a sponge. This must be removed and replaced with a small muslin bag filled with moist oxide of iron—new purifying material will do very well. There is some little

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doubt as to the perfect safety of this form of respirator, as possibly some little sulphuretted hydrogen passes through the moist oxide of iron ere it becomes active, and this may prove a source of some little danger.

But whatever doubt there may be of the efficiency of the above respirators, there is no doubt whatever of the efficiency of the apparatus made by the same firm and shown in Figs. 117 to 120. The canvas helmet is placed over the head as shown in Fig. 117; the foot bellows, Fig. 118, or the hand bellows, Fig. 119, is put in the pure open air and connected by means of the armoured india-rubber tube, Fig. 120, to the tube at the



FIG. 115.—Aluminum Respirator.



FIG. 116.—Universal Valve Respirator.

back of the helmet. The current of cold air passes over the face of the wearer of the helmet, sweeping away all poisonous gases before it, and so long as the bellows are at work the man is absolutely safe!

### *Treatment of Persons accidentally poisoned by Sulphuretted Hydrogen, etc.*

A very useful notice as to what is to be done under such circumstances is issued in sheet form, suitable for framing, by the proprietors of "The Chemical Trade Journal" (Messrs Davils Bros.).

A copy of this should be put up in a convenient part of the works.

### *Sulphuric Acid*

The carriage of this material *in carboys* should only be adopted as a last resource, as the writer knows of no more disagreeable task than unloading a truck containing carboys of acid, when one or two have been broken in transit, and, however carefully the packing may be done at the acid works, breakages in transit are of frequent occurrence. When, how-

ever, acid *must* be taken in carboys, look well after the empties, as neglect of this leads to much friction between the maker and consumer of the acid, and the makers have certainly much cause for complaint in this direction.

Where a railway siding runs into the works and the quantity of acid consumed warrants it, it is best to have the acid sent



FIG. 117.

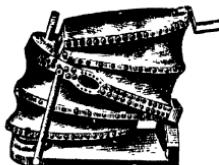


FIG. 118.



FIG. 119.

ZIMMER'S SAFETY HOOD RESPIRATOR.



FIG. 120.

in steel railway tanks holding about ten tons each. In this case a storage tank made of wood lined with 8 to 10 lb. lead should be provided and fixed in such a position that the acid may be run into it from the railway tank by gravitation. It should be sufficiently large to allow the whole of the contents of the railway tank to be run off into it at the operation, as to partially empty the railway tank and then shut it up and down, often with the man-lid insecurely fastened, is disastrous to the well-being of the tank. A storage tank 10 feet  $\times$  6 feet  $\times$  4 feet

will comfortably hold ten tons of acid. From this tank it is elevated to the tank for supplying the saturator, either by means of an acid elevator similar to that shown in Figs. 30 and 31, or by a steam regulus injector. And here the writer would like to put in a plea for the acid maker with regard to the detention of the railway tanks. These tanks are *not intended to act as acid stores*, and for a consumer to keep them under load day after day while the maker is pressed for them to supply his orders is decidedly unfair.

The acid should be sampled on receipt, the best way to do so being by taking the man-lid off the tank and dipping a long glass tube  $\frac{1}{2}$ -inch in diameter right through the contents (except the little dirt that accumulates at the bottom of the tank), using the tube as a pipette. Get the acid to  $60^{\circ}$  Fahr. and test with the hydrometer. Should it show too low, before complaining check the correctness of the hydrometer by means of the specific gravity bottle. It will often be found that the hydrometer and not the acid is at fault. If acid is kept for some time a sample taken from the bottom of the tank will often show much higher than if taken from near the surface. The writer remembers a case where a complaint was made about the strength of some acid, and on the acid-maker's chemist being sent over to investigate it he discovered that the sample had been taken from the surface of a tank where it had been stored for about two months, and further that the tank was situated in such a position that rain readily blew into it. Comment is unnecessary.

Where the acid is supplied by a firm within carting distance of the consumer's works it may conveniently be brought in steel barrels holding either about 25 cwts., or 2 tons 10 cwts. each, and discharged into a similar (but not necessarily so large) tank as that suggested for that received by rail.

A ton of sulphate of ammonia requires about 14.84 cwts. pure acid (= 18.32 cwts. at  $144^{\circ}$  Tw.) for its manufacture.

#### *Ammoniacal Liquor*

To ascertain the quantity of ammoniacal liquor required, roughly, to make a ton of sulphate of ammonia, divide 25.75 by the percentage of total  $\text{NH}_3$  contained in the liquor. The answer will be the required quantity expressed in tons. To ascertain the actual loss of ammonia in working, a small sample of the ammoniacal liquor that is being used should be taken at regular intervals, say every six hours, and placed in a Winchester quart bottle, provided with an india-rubber stopper, and at the end of each week an average sample taken from this, and tested for total  $\text{NH}_3$  as previously described. If the tons

of liquor consumed be multiplied by the figure and the result divided by the actual percentage of ammonia contained in the sulphate of ammonia delivered during the week, the result will show the quantity of sulphate of ammonia producible, from which deduct the amount actually produced, and the difference will represent the loss.

(Should the quantity of mother liquor in stock at the close

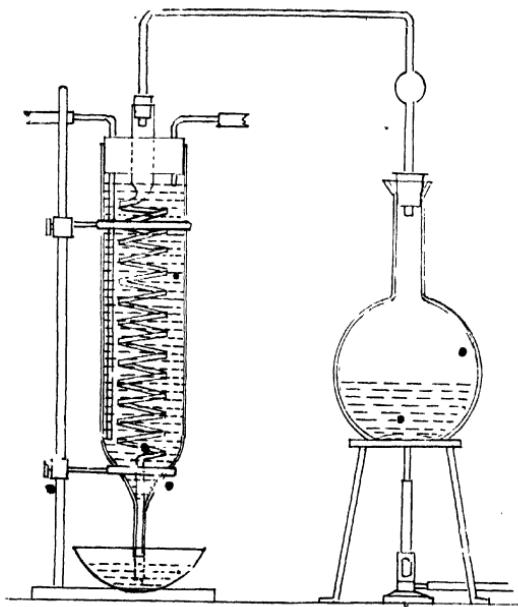


FIG. 121.

of the week be greater than at the beginning, it is obvious that this will account for some of the apparent loss.)

#### *Analysis of Waste (or Spent) Liquor*

In a large works, samples of the waste liquor as it leaves the still are usually taken every half hour and placed in 6-oz. corked bottles. Should a sample smell of ammonia, the bottle is marked with a cross in chalk, and this particular sample is then tested separately. As each bottle is labelled with the time the sample was taken or placed in a particular order,

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should the suspected sample test high, the time can be located and the matter taken up with the man in charge. To reduce the number of tests the remainder of the batch for each 8 or 12-hour shift may be mixed to form one sample.

The samples may be tested for total  $\text{NH}_3$  in the same manner as that described in Chapter II. for testing ammoniacal liquor, except that 100 c.c.'s of the waste liquor and only 5 c.c.'s of the normal sulphuric acid are taken, this, of course, modifying the calculation accordingly.

Or, if preferred, the apparatus shown in Fig. 121 may be used, in which case the distillation is made into distilled water, and titration with normal sulphuric acid only is required.

In good working the total ammonia should not exceed 0.02%.

To test for the free lime, shake up the average sample thoroughly, take 100 c.c.'s, boil in a beaker to expel the ammonia, and then titrate with normal oxalic acid solution (made by dissolving 63 grammes of the crystals in water and making up the bulk to 1 litre), using a solution of phenolphthaleine as the indicator. 1 c.c. would equal 0.02% calcium or 0.028% calcium oxide. A suitable quantity of free lime would be, say, from 2 to 5%.

## APPENDIX

### FEATURES IN DESIGNING A SULPHATE OF AMMONIA HOUSE

An article entitled as above by Mr Frederick Shewring is here reproduced, with additions, from *The Gas World*, with the author's acknowledgments to Mr Shewring and the publishers of *The Gas World*.

It does not fall to the lot of every engineer and manager of gasworks to have to build a sulphate of ammonia house and instal the necessary plant. For this reason it is possible that this subject has not had the same amount of consideration as is apt to be devoted to the design or erection of the various other plant found on a gasworks. It is observed on some works that the stills, heater and saturator are housed in some makeshift building, and it is perhaps not until the apparatus has to be overhauled, or improvements added to procure efficiency, that the drawbacks of the situation become obvious.

There are a few outstanding details which command attention, when designing a new building to take new manufacturing plant, and although they may have to be varied or modified in some instances to meet special local conditions, it is well to have them under consideration beforehand, and endeavour to take advantage of any feature of convenience. The writer has in mind a case where a scaffold or legs have to be employed each time to enable the sections of the stills to be taken apart. With a little forethought, at the time when the building was erected, provision could easily have been made for a permanent structure to have been added to attach a set of lifting tackle, without incurring extra expense, simply by the rearrangement of the rolled steel joists which support the crude ammoniacal liquor storage tank in the building. It is always well to bear in mind that, owing to the destructive effect of any sulphuric acid that may leak through the draining floor, or from the acid tank, the portions of the plant that are likely to be affected from such cause, such as, for instance, a chimney-stack (if steam happens to be generated in the building), stills or overhead tank supports should be kept as far as convenient from the vicinity of acid storage accommodation.

#### *The Site*

Any special advantages that the site affords for attaining economy in matters of unloading acid tanks, supplying the ammoniacal liquor to stills, or feeding the saturator direct with acid without the employment of mechanical means are

features that should receive due consideration. The loading of the sulphate from a higher level into carts or railway trucks saves cost in handling, and these matters materially assist in making the manufacture of sulphate of ammonia more remunerative. It is really a question of levels. It may not prove advantageous to specially provide for all these conveniences on some sites, but with a little scheming and judicious expenditure, the costs can, under certain circumstances, soon be recouped.

#### *Chimney Flue and Stack*

It is more costly to instal a steam-raising plant solely for use in sulphate manufacture than to obtain a supply of steam from the existing plant found on all gasworks, other than the very smallest. It takes a certain amount of the attendant's time to look after the firing of the boiler, and this time could often be more profitably employed attending to the working of the plant. If, however, there is no alternative but to include a boiler in the sulphate house, the question of providing a chimney-stack and its position are features to be considered.

The idea, that has been put into practice, of running the flue from steam boiler to chimney alongside of the drying store, with the object of furnishing heat to assist the drying of the sulphate, is excellent, if precautions are taken to rigidly prevent the mother liquor from gaining admittance into the brick-work or flue. Artificial means of drying the sulphate are essentially convenient at times, more particularly on works which have not a centrifugal drying machine. The writer does not, however, like the practice of forming the chimney-stack on the corner walls of the building, unless the house is of large dimensions. Even in this case, the walls that form the back of the drying store should obviously be avoided. The erection of the chimney-stack a short distance from the sulphate house is preferable in the majority of cases, and any underground acid storage tanks that may have to be provided should be kept at a reasonable distance away, consistent with the convenient handling of the acid. The writer has an instance in mind where a chimney-stack was almost ruined owing to acid finding its way into the foundations.

#### *The Floor Level*

The floors of sulphate houses, apart from drying store, are, almost without exception, kept on one level; but if the effluent liquor, or a portion of it, is intended to be used for lime-slaking or lime-mixing purposes, it is advisable to fix the liming tanks at a lower level than the stills, to enable a feed of effluent to gravitate to the liming tanks as required. This will save employing an injector, steam or hand pump for lifting the liquor,

which will prove a source of convenience. The floor space where the liming apparatus is to be fixed should be 2 feet lower than the remainder of the floor of the building. If the mixing tank is then let into the ground another 12 inches, the slaking tank can be fixed sufficiently low to enable the spent liquor running from the secondary still to gravitate to it.

It may be argued that the stills themselves could be fixed on a brick or concrete foundation a couple of feet above floor level, and there would then be no necessity to excavate the ground especially to accommodate the liming apparatus. This arrangement would quite meet requirements; but it is by far the most costly method to adopt. The height of building would correspondingly be increased.

The floors of sulphate houses may be formed of asphalt, or bricks laid without cement or mortar jointing, in preference to a cement flooring. The former methods can more easily be re-levelled and repaired as occasion demands.

#### *The Draining Floor*

The chief points to aim at in constructing the draining floor is to see that sufficient fall of floor is provided to the mother liquor well; also, that the bearer joists are deep enough to prevent the mother liquor saturating the sulphate in the event of the sheet-lead lining becoming buckled, and to guard against the possibility of the drainings leaking into the floor or brick-work of the building. Fig. 1 shows the proposed method of laying the bearer joists with the object of collecting the drainings to one well, provided near the centre line of the draining floor. The lead-lined floor, from the walls to centre, should be laid sloping, with a  $1\frac{1}{2}$ -inch fall for every 8 feet run of flooring. The bearer joists or floor board supports, which are laid loose on the sheet-lead, should be 4-inch by 3-inch or 5-inch by 3-inch red deal. By having the joists 4 or 5 inches deep, provision is made for the acid to flow freely to the well, even in the event of a lifting action of the sheet-lead taking place, as is known to happen frequently.

The height to which the sheet-lead may be carried on the side walls of the buildings is usually governed by the depth of floor space allotted for storing the sulphate. If the front part of the storage accommodation is not boarded up, the width of floor from back to front may be taken as the height to which the sheet-lead may be extended up the walls. The sides of wall should afterwards be metalled and. The writer does not think that any advantage is gained by fixing a wooden framework around the walls on which the sheet-lead is attached, although some engineers prefer it. For securing the top of sheet-lead to walls, 4-inch by 3-inch wooden blocks should be

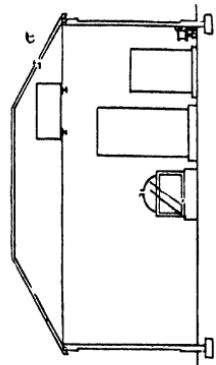


Fig. 3.  
H, Mother liquor well.

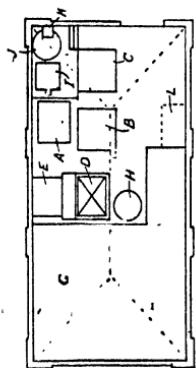


Fig. 2.  
A, Heater. B, Primary Sull. C, Secondary Sull. D, Saturator. E, Draining table. F, Pump, etc. G, Store room. H, Mother liquor well. I, Lime-making tank. J, Lime-making tank. K, Pump, etc. L, Weighing machine.

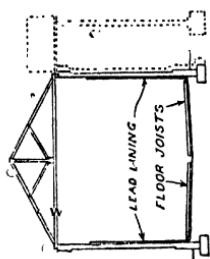


Fig. 1.  
A, Heater. B, Primary Sull. C, Secondary Sull. D, Saturator. E, Draining table. F, Pump, etc. G, Store room. H, Mother liquor well. I, Lime-making tank. J, Lime-making tank. K, Pump, etc. L, Weighing machine.

provided to catch any acid or mother liquor that may overflow or leak from the saturator. If this is omitted, the flooring after becoming saturated with acid will readily lift the said saturator, and

built into brickwork, and 3-inch by 2-inch or 4-inch by 2-inch horizontal members attached thereto should be extended around the storage floor. The sheet-lead can then be dressed over this and suitably secured.

It is a wise precaution to give several coatings of tar on the brickwork around the sulphate storage room, and in fact the whole interior of the building may be treated if desired in a similar way. The writer has found that after the application of several coats of boiled tar and pitch mixed, the brickwork resists the destructive nature of the weak acid in the sulphate. His tests have not yet been carried out over a prolonged period, but he is convinced of its suitability for the purpose referred to.

With the open type of sulphate storage floor, as illustrated, care must be exercised to prevent the acid from draining over the front end of the flooring. The sheet-lead can be stiffened along the front by dressing it to double thickness, and if this is kept 1 inch higher than the flooring boards and the same distance from the end of boards, no trouble will be experienced.

It may be desirable to board up, for a few feet, a portion of the front of the storage space, to add somewhat to the storage capacity. This must not be extended too far, because it will be found inconvenient when the bagging of sulphate takes place.

Underneath the saturator a sheet-lead tray should be provided to catch any acid or mother liquor that may overflow or leak from the saturator. If this is omitted, the flooring after becoming saturated with acid will readily lift the said saturator, and

possibly cause the connections or flanges of the pipes to give. The lead tray should be laid with a fall to the mother liquor well, and a pipe or outlet passage arranged to convey the liquid thereto.

#### *Arrangement of Plant*

A glance at Figs. 1, 2 and 3 shows the suggested arrangement of plant. It will be noticed that provision has not been made for the inclusion of steam-raising plant in the building. If a steam boiler has to be installed a lean-to building could readily be erected at the end of the house near the doorway; or, of course, the boiler could be housed inside the building and the accommodation for storing the sulphate thereby reduced.

It will be observed that one of the rolled steel joists supporting the liquor tank is arranged centrally over the primary still and the superheater. Lifting blocks can be attached to this, and it will prove useful when taking the plant apart. If the brickwork opposite the centre of the secondary still is corbelled out sufficiently to form a bearing for a rolled steel joist, or, better still, if an iron shoe is built into the brickwork, the other end of the joist can easily be secured by a support hanging from the girder which carries the ammoniacal liquor tank. This is not costly, and, when once provided, it is always available to meet emergencies.

If for any specified reason it is desirable to erect the ammoniacal liquor tank outside the sulphate house, it can be carried out by somewhat strengthening the wall of building and resting the end or side of tank thereon, and supporting the remainder on a couple of iron, or steel columns. The tank should be covered in to prevent waste of ammonia. The dotted lines in Fig. 1 explain these remarks.

Another method of making provision for the taking apart of sections of the stills in the event of cross girders being absent is to fix in the walls directly overhanging the plant a steel girder bracket for lifting tackle to be attached.

#### *Loading the Sulphate of Ammonia*

The loading by manual labour of 2-cwt. bags of sulphate on drays or into railway trucks is arduous work and somewhat costly. With plants turning out a large tonnage of sulphate, it has occurred to the writer that arrangements may be made for an adaptation of the monorail or telpher system, operated by hand or steam rope-winding machine to enable the bags of sulphate to be taken out of the sulphate house and placed direct into carts or railway waggons. It is just a question whether the monetary saving in labour would justify the initial outlay. Each case would have to be considered from its own particular

standpoint. If the loading floor is such that a dray can back aginst it, and the sulphate can be wheeled on sack trucks direct on to lorries, without having to lift the sacks of material several feet, much of the difficulty will be overcome. The provision of a light jib crane for loading purposes would, in many instances, prove to be a profitable investment. The position of the site may be helpful in determining the most advantageous arrangement to instal. It is certainly an important item, when it is remembered that it takes three men to lift a 2-cwt. bag of sulphate on to a railway dray, besides the carter who receives the bag on the lorry.

*Details of Construction*

The roof trusses, if constructed of wood, cost less in upkeep, and for this reason the writer prefers them to steel or ironwork. The type of truss to employ is dependent upon the width of

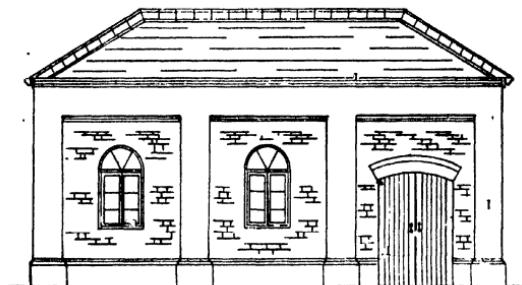


FIG. 4.—Sulphate House. Elevation.

building. If the width is 30 feet or less, a king-post roof truss, as illustrated in Fig. 1, can be adopted. If the building is over 30 feet, a queen-post truss should, for preference, be used. In deciding upon the size of members for a given roof-truss, or, in fact, for any other type of construction, always endeavour to employ material of similar size or pieces cut from multiples thereof. For instance, assuming that the king-post referred to is for a span of about 24 feet, it can be made up of the under-mentioned members:—tie beam 8 inches by 4 inches, struts 4 inches by 4 inches, king-post 6 inches by 4 inches, principal rafters 4 inches by 4 inches, and the purlins 8 inches by 4 inches. The weight of the roof the framing is called upon to support has, of course, a bearing on the dimensions of its members. The writer rather favours the use of cement-asbestos shingles or corrugated sheathing for roofing buildings of the class under consideration.

Figs. 4, 5 and 6 show, elevation, section, and roof plan

of a good-class building, the size of which can be modified to meet the requirements of large or medium-sized gasworks. The door is shown at the front of building, but it can, if more convenient, be placed at the one end, as shown on

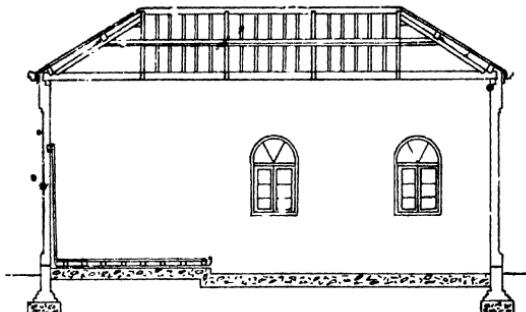


FIG. 5.—Sulphate House. Section.

Fig. 2. If the position of the door is altered another window could be added.

The following is an abbreviated specification for the sulphate house structure:—

*Foundations.*—Excavate soil 2 feet wide to required depth and ram solid. Lay a bed of concrete 18 inches deep.

*Floor.*—The interior of the building is to be excavated to



FIG. 6.—Sulphate House. Roof Plan.

a depth of 6 inches. Ram solid and fill in with concrete. When set, lay in sections an inch bed of cement, 2 parts, sharp gravel sand, 2 parts, and fine chippings, 1 part, thoroughly mixed and floated to a true level.

*Concrete* to be composed of 6 parts broken brick or ballast to pass through 1½-inch mesh and 1 part each of sharp sand and approved Portland cement.

*Brickwork*.—Build footing as shown on drawings. (These can be omitted on good foundations.) Build piers in 18-inch brickwork to a height of 2 feet above ground level and reduce to 14-inch brickwork. The panel walls to be 5½ inches reduced to 9 inches. Two plinth courses to be laid to form the 4½-inch set-offs. Blue bricks can be used if desired. The brickwork is to be "Old English bond," alternate layers of headers and stretchers.

*Damp-proof Course*.—Lay a course of slates in cement throughout the thickness of wall, position as shown on drawing.

*Woodwork* throughout to be of best red deal, well seasoned and free from defects.

*Wall-plate* to be 4½ inches by 3 inches.

*King-post Trusses*.—Construct to dimensions and size previously given. Strengthen king-post to tie-beam with wrought-iron stirrups with gibs and coppers and principal rafters to tie-beam with wrought-iron stirrups. (Stirrups 2-inch by ½-inch flat iron.)

*Cleats* to be provided for purlin support as shown.

*Pole-plates* to be 4 inches by 4 inches.

*Common Rafters* to be 4 inches by 2 inches, fixed 15 inches centre to centre.

*Ridge Boards* 9 inches by 1½ inches.

*Slating*.—Lay best quality Duchess slates (24 inches by 12 inches) centre nailed with a uniform lap of 4 inches and 10-inch margin. Double courses to be provided at the eaves. The underside of roofing to be torched. Battens 2 inches by 1 inch. If cement asbestos shingles are preferred, specify to be laid diagonally and in accordance with the maker's specification.

*Roof Ridges*.—Blue ridge tiles to be securely fixed on ridge boards in neat cement. If lead work is desired, specify for a 2-inch roll to be spiked on ridge boards and dressed with 6 lbs. sheet-lead. Lead tingles 18 inches long are to be provided 4 feet apart under the hip rolls, and the sheet-lead is to be fastened thereto.

*Facia Board* to be 9 inches by 1 inch, with beaded edge.

*Guttering and Down Spouts*.—5-inch O.G. guttering to be fixed around building with suitable fall to the two 4-inch down spouts. Down spouts with feet to be provided and fixed in approved position.

*Windows*.—Provide and fix cast-iron window frames as required, and glaze on completion of work with 26-ounce glass.

*Door (hanging type)*.—To be made in halves. The framing to be constructed of 2½-inch by 7-inch hanging and meeting stiles, and 1½-inch by 7-inch bottom, middle, and top rails; and afterwards board the entire height of door with 1-inch

match boarding. Paint door with priming and two finishing coats in approved colour. Bolt to the doors four wrought-iron strap-hangers, 3 inches by  $\frac{1}{2}$  inch, and drill and fix  $\frac{1}{2}$ -inch centre pins for the 4-inch diameter cast-iron carrier wheels to work thereon. Provide and fix runner rail of sufficient length to enable the door to open to full width of doorway.

Fig. 7 shows details of construction for a ventilating louvre, suitable for the sulphate house, should it be deemed advisable to add this.

#### *Cooling Plant*

In passing, brief reference may be made to the outside condensing plant. Whatever type of plant is installed to cool the

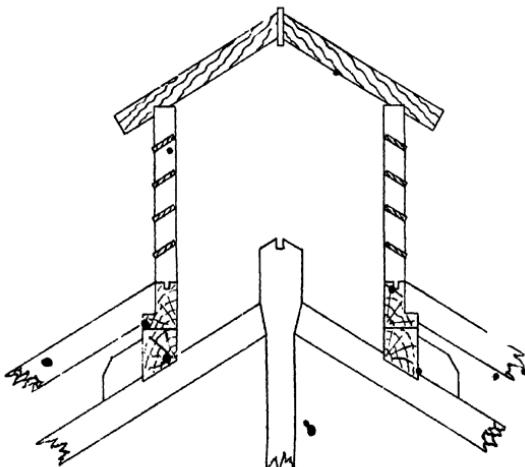


FIG. 7.—Ventilating Louvre.

waste gases, provision must be made for readily cleaning out the apparatus. If this is neglected stoppages will be frequent, for it is found on many plants that ammonia at times travels forward with the waste gases, and carbonate of ammonia deposits in the condensing plant. This solidifies when the sulphate plant is shut down and the pipes become cool. When the substance referred to is removed, it is black in appearance, due to discolouration by hydrocarbons passing forward with the waste gases. The arresting of lime sludge is of real importance, for if the spent liquor is allowed to pass forward through pipes direct to sewer or other outlet, without the removal of the lime sludge, a hard substance will form in the pipes and eventually block

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up the passage. The writer remembers examining on one occasion a 4-inch glazed pipe which was completely choked with lime sludge, and it had become so hard that a number of pipes were broken in the endeavour to dislodge the incrustation.

The object of this article is not altogether so much the describing of a model sulphate of ammonia factory, because the writer is perfectly aware that what is admirably suited on some works is quite out of place at others ; but the suggestions made may, the writer hopes, be used to advantage by some members of the profession who have to deal with work of this class.

### COMPARISON OF AMMONIACAL LIQUORS FROM VARIOUS SOURCES

From	% NH <sub>3</sub>			% H <sub>2</sub> S	% CO <sub>2</sub>
	Free	Fixed	Total		
Gasworks . . .	1.572	0.229	1.801	0.234	1.875
Coke Oven Plants . .	0.975	0.198	1.173	0.130	1.301
Shale Works . .	0.573	0.036	0.609	0.026	0.938
Blast Furnaces in Iron Works	0.257	0.035	0.292	Nil	0.526

Averages taken from Annual Report for 1896 of the Chief Inspector under the Alkali, etc., Works Acts. For further particulars see this Report.

### COMPARISON OF THREE ANALYSES OF AMMONIACAL LIQUOR MADE BY THE WRITER DURING YEAR 1899

Sample No.	% NH <sub>3</sub>			% H <sub>2</sub> S	% CO <sub>2</sub>	°Tw. at 60° F.
	Volatile	Fixed	Total			
1	2.3060	0.6459	2.9519	0.536	2.454	6.7
2	1.7232	0.5456	2.2688	0.530	1.841	4.8
3	1.7580	0.6103	2.3683	0.442	1.966	5.8

1 and 3 were from the same gasworks, and 2 from another gasworks, all in the same city.

## SULPHURIC Acid

The following Table shows the quantity of pure Sulphuric Acid ( $H_2SO_4$ ) contained in 100 parts dilute at different densities, according to Dr Ure's Dictionary.

(Specific gravity of water = 1.000)

$H_2SO_4$	Spec. Grav.	$^{\circ}Tw.$ at $60^{\circ}F.$	$H_2SO_4$	Spec. Grav.	$^{\circ}Tw.$ at $60^{\circ}F.$	$H_2SO_4$	Spec. Grav.	$^{\circ}Tw.$ at $60^{\circ}F.$	$H_2SO_4$	Spec. Grav.	$^{\circ}Tw.$ at $60^{\circ}F.$
100	1.8460	169.2	75	1.6500	130.0	50	1.3884	77.7	25	1.1792	35.8
99	1.8438	168.7	74	1.6415	128.3	49	1.3788	75.7	24	1.1706	34.1
98	1.8415	168.3	73	1.6321	126.4	48	1.3697	73.9	23	1.1626	32.5
97	1.8391	167.8	72	1.6204	124.1	47	1.3612	72.2	22	1.1549	30.9
96	1.8366	167.3	71	1.6090	121.8	46	1.3530	70.6	21	1.1480	29.6
95	1.8340	166.8	70	1.5975	119.5	45	1.3440	68.8	20	1.1410	28.2
94	1.8288	165.7	69	1.5868	117.3	44	1.3345	66.9	19	1.1330	26.6
93	1.8235	164.7	68	1.5760	115.2	43	1.3255	65.1	18	1.1246	24.9
92	1.8181	163.6	67	1.5648	112.9	42	1.3165	63.3	17	1.1165	23.3
91	1.8120	162.5	66	1.5503	110.0	41	1.3080	61.6	16	1.1090	21.8
90	1.8070	161.4	65	1.5390	107.8	40	1.2999	59.9	15	1.1019	20.4
89	1.7986	159.7	64	1.5280	105.6	39	1.2913	58.2	14	1.0953	19.0
88	1.7901	158.0	63	1.5170	103.4	38	1.2826	56.5	13	1.0887	17.7
87	1.7815	156.3	62	1.5066	101.3	37	1.2740	54.8	12	1.0809	16.1
86	1.7728	154.0	61	1.4960	99.2	36	1.2654	53.1	11	1.0743	14.8
85	1.7640	152.8	60	1.4860	97.2	35	1.2572	51.4	10	1.0682	13.6
84	1.7540	150.8	59	1.4760	95.2	34	1.2490	49.8	9	1.0614	12.3
83	1.7425	148.5	58	1.4660	93.2	33	1.2409	48.2	8	1.0544	10.9
82	1.7315	146.3	57	1.4560	91.2	32	1.2334	46.7	7	1.0477	9.5
81	1.7200	144.0	56	1.4460	89.2	31	1.2260	45.2	6	1.0405	8.1
80	1.7080	141.6	55	1.4360	87.2	30	1.2184	43.7	5	1.0336	6.7
79	1.6972	139.4	54	1.4265	85.3	29	1.2108	42.1	4	1.0268	5.3
78	1.6860	137.2	53	1.4170	83.4	28	1.2032	40.6	3	1.0206	4.1
77	1.6744	134.9	52	1.4073	81.4	27	1.1956	39.1	2	1.0140	2.8
76	1.6624	132.5	51	1.3977	79.5	26	1.1876	37.5	1	1.0074	1.5



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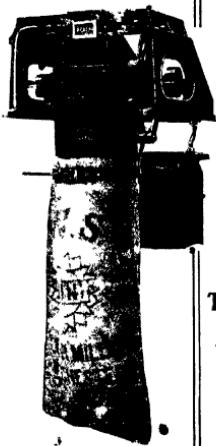
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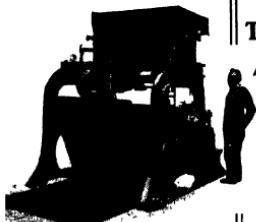
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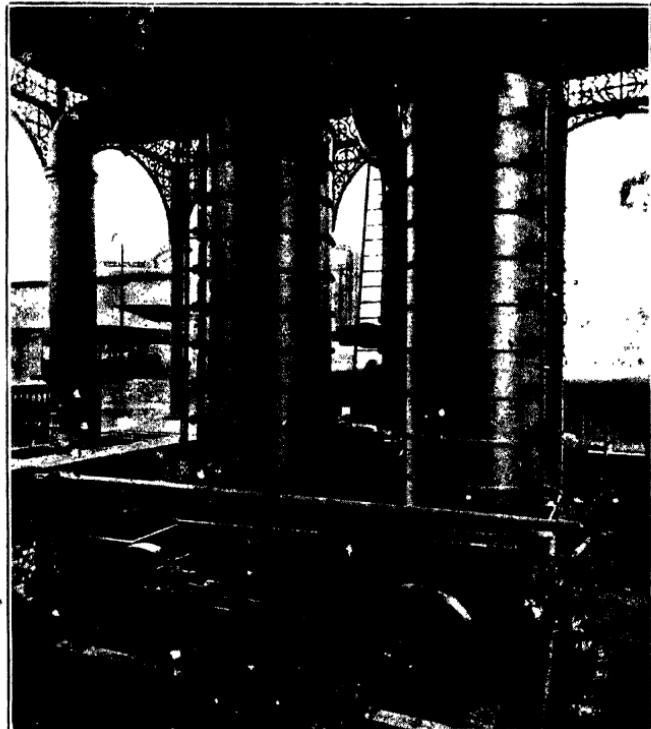
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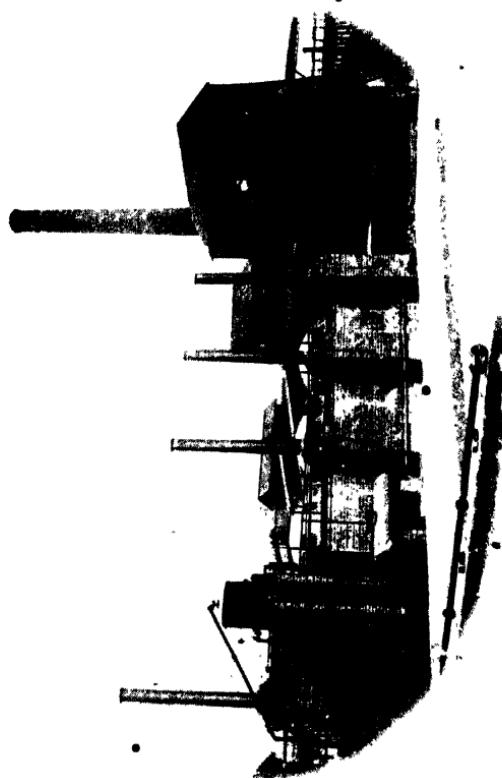
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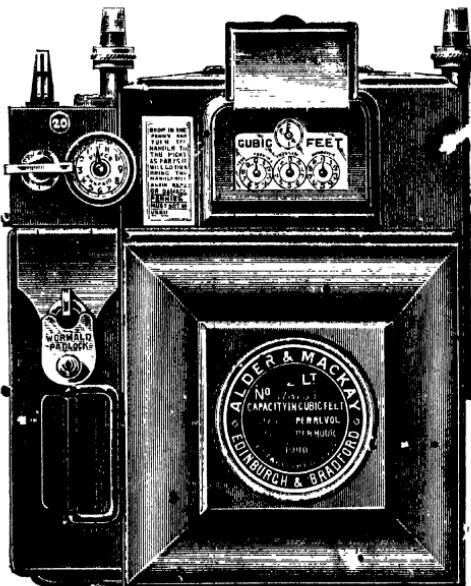
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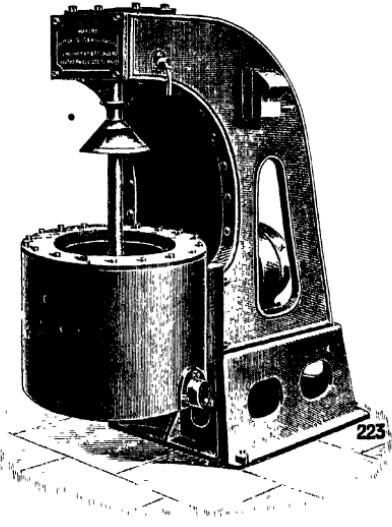
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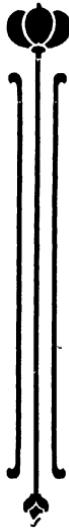
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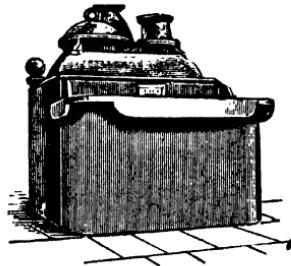
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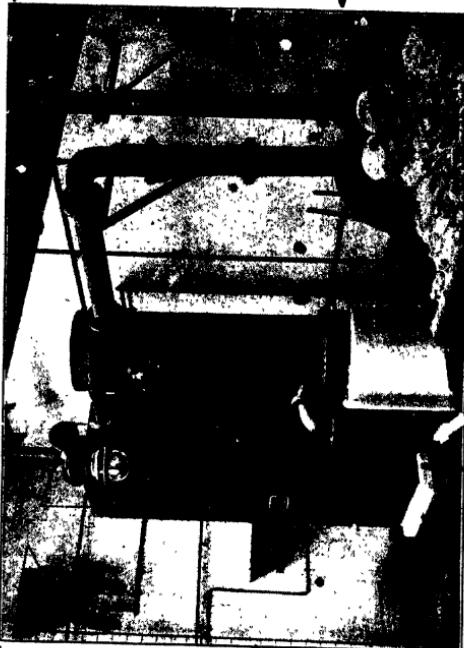
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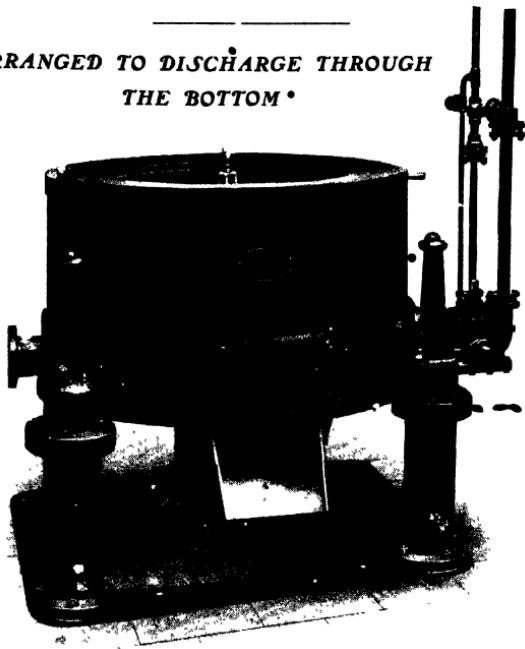
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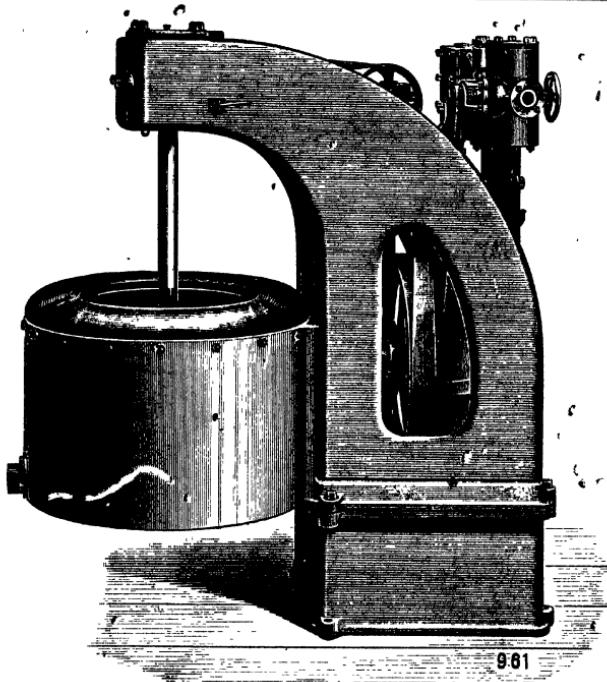
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